NORMAN A. JOHNSON
Editor

CHARLES A. WHITEHEAD
Managing Editor

MYRON D. REESER
Business Manager

HERBERT A. STAUDERMAN
Advertising Manager

JAMES A. DOYLE
Promotion Manager

FRANK R. MURPHY
Production Manager

LILLIAN M. THOMPSON
Circulation Manager

AMERICAN
DYESTUFF REPORTER

Devoted to Textile Chemistry
BLEACHING • DYEING • PRINTING • FINISHING

OFFICIAL PUBLICATION OF THE PROCEEDINGS AMERICAN ASSOCIATION OF TEXTILE CHEMISTS AND COLORISTS

American Dyestuff Reporter was founded in 1917 and the Textile Colorist and Converter (formerly Textile Colorist) was incorporated with it in January, 1949. Published every other Monday by the HOWES PUBLISHING COMPANY, INC., 44 E. 23rd Street, New York 10, N. Y. C. R. Howes, President; Ernest J. Finan, Executive Vice-President; Norman A. Johnson, Vice President & Treasurer; Myron D. Reeser, Vice-President; C. Essenbreis, Secretary.

Telephone SPring 7-9364

Contents

VOLUME 43 OCTOBER 25, 1954 NUMBER 22

• *Special Articles*

Barotor Loading Improvement 709
Paul M Cole

Historical Notes on the Wet-Processing Industry. VI—The Dual Life of Edward Bancroft 712
Sidney M Edelstein

• *Proceedings of the American Association of Textile Chemists and Colorists*

FEATURE ARTICLES

A New Method of Vat-Dyeing Knit Goods P719
Robert D Blum, Jr

A Review of Textile-Shrinkage Control Processes and Test Methods P726
John F Warner

Vat Dyes—A Miscellany P730
Maurice R Fox

MISCELLANEOUS

AATCC Calendar P715
Activities of the Local Sections P715
Research Newsletter #17 ..... P716
Theme Selected for Perkin Centennial P717
Employment Register P717
Auditor's Report P717
Treasurer's Report P718

• *Departments*

Patent Digest 714
Paul Wengraf

Abstracts 733
News of the Trade 736
General Calendar 740

Copyright, 1954, Howes Publishing Co, Inc

Contents of previous issues can be found by consulting the Industrial Arts Index and the Engineering Index, Inc, at your library

ISOLANS*

new metal complex colors

BRIGHTER

MORE SOLUBLE

BETTER LEVELING

FOR WOOL • SILK • NYLON

*Trade Mark Applied For

DYESTUFFS INTERMEDIATES

VERONA

TEXTILE AUXILIARIES

Trademark Reg. U.S. Pat. Office

DYESTUFFS

A DIVISION OF VERONA CHEMICAL CO.

SPRINGFIELD ROAD, UNION, NEW JERSEY

Branches: Ardmore, Pennsylvania • Providence, Rhode Island • Rock Hill, South Carolina

AMERICAN
DYESTUFF REPORTER

VOLUME 43 OCTOBER 25, 1954 NUMBER 22

BAROTOR LOADING IMPROVEMENT

PAUL M COLE
E I duPont de Nemours & Co Inc

A new method of loading Barotor dyeing machines to permit use of random piece lengths is described. This improvement is said to provide added versatility and greater economy.

The fabric is processed as a batch without separation into individual pieces and the total length can be anything up to the maximum multiple layer capacity of the rotor. Existing Barotors can be adapted readily to the new system with the substitution of a plaiter and scray for the present loading creel.

Other Barotor improvements, some of which are already in commercial use, are listed in detail. These include rotor design simplifications and automatic slack measurement.

INTRODUCTION

THE Barotor, a pressurized piece goods dyeing machine, was first disclosed to the trade by the Du Pont Company in August of 1952 *(1)*. Continued research led to design simplifications that were announced in May of 1953 *(2)*. The machine up to that point necessitated the matching of piece lengths in each batch, a practice that limited the flexibility of use. This condition of operation was imposed by a parallel type of loading in which a number of pieces were fed simultaneously from a creel to the large rotor. Accordingly, the initial Barotors introduced in the trade necessitated special handling to form batches of nearly equal lengths.

A new development called "series loading" has now obviated the previous shortcoming by providing for the handling of random lengths of fabric sewed end to end into continuous yardage up to the maximum multiple layer capacity of the rotor. Practically no change is required of the machines presently in use, other than auxiliary equipment. The present loading creel merely has to be replaced with a fabric preparation assembly consisting of a modified plaiter and a scray. The first Barotor to be so equipped, as shown in Fig 1, is installed at Du Pont's Textile Research Laboratory.

DISCUSSION

Perhaps the best way to convey an understanding of the new loading practice is to point out that, if a continuous length of material could be wound in a number of turns on the 100-yard periphery of a cylinder almost one hundred feet in diameter, this large multiple-layer loop could be slipped off the cylinder and a portion of it carried over the rear head of the rotor. Loading of the inner bars in the usual manner would then bring the entire batch of material into the normal operating position on the rotor. Obviously, such a cylinder is far too large for practical use, but the same thing can be accomplished in compact space with a special plaiter and scray.

The operations involved, other than the usual dyeing or chemical treatment of the fabric, are as follows:

1) winding
2) Barotor loading
3) Barotor unloading
4) unwinding

Steps (1) and (4) do not involve the Barotor directly, and they can be performed in a different mill area or carried out on separate batches in the vicinity of the Barotor while the machine is in use. In the latter case, elements of the loading equipment can be employed for all four operations. Steps (2) and (3) are parts of the dyeing cycle, but they require less time now than formerly.

FABRIC PREPARATION——The first step starts with the material in a continuous roll on the supply mandrel. (See Figure 2). The leading end of the fabric is threaded through a plaiter and fed into the top of a scray, the lower rolls of the plaiter being held in a fixed position at an extreme angle of swing. When a length equal to the known dis-

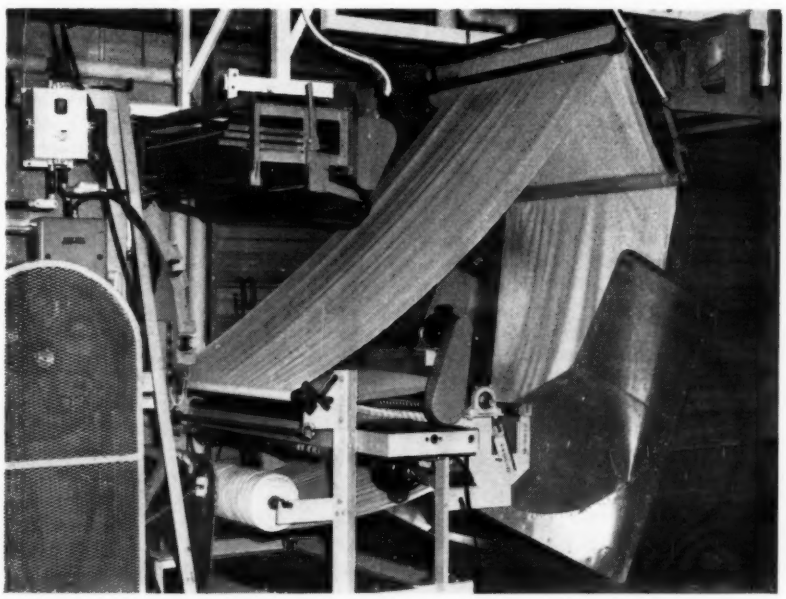


Figure 1

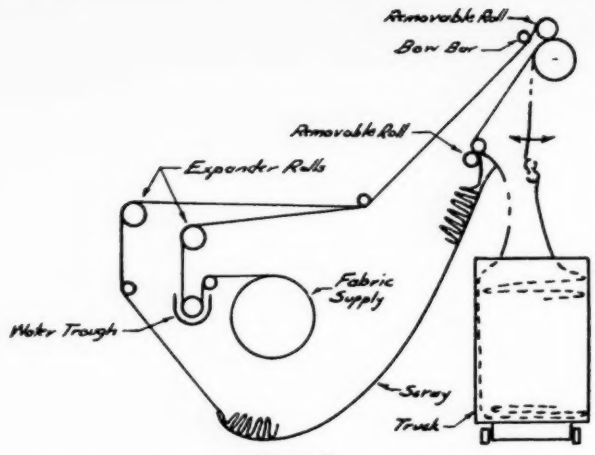


Figure 2

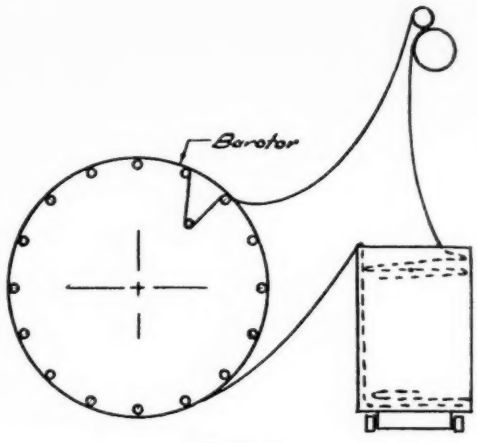


Figure 3

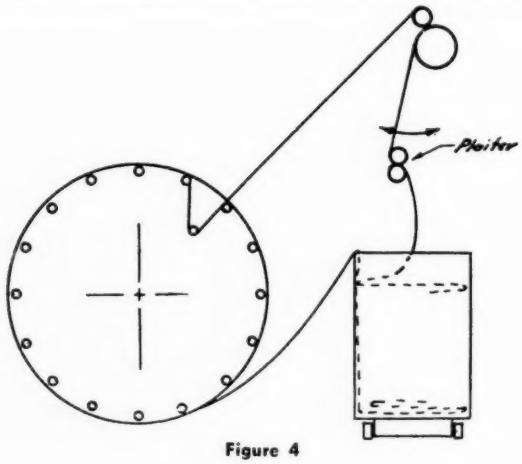


Figure 4

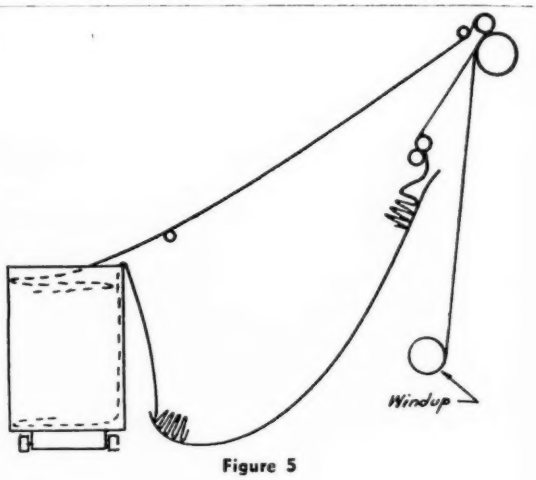


Figure 5

tance of the fabric path of one wrap on the Barotor (approx 100 yds) has been unwound, the leading end is withdrawn from the scray and tacked to the portion coming from the supply roll. Preferably, a leader should be employed so that the joining takes place at the junction of two pieces. The sewing can then be made completely across the fabric, instead of just at the selvages. The large loop thus formed then repeats its course through the plaiter and scray, thereby causing a second layer of equal yardage to be added to the first. Adherence of the fabric layers is greatly enhanced by a film of water. When dyeing follows a continuous boil-off without heat setting, the fabric will already be wet. When heat-set material or fabric in the greige is to be loaded on the Barotor, it should be run through a water trough. Additional water can be sprayed in the scray and the lower part of the scray can be submerged to assure complete wetting. This operation is continued with the winding of more and more layers until the material left on the supply mandrel is sufficient for just one more layer. The plaiter rolls are then put into normal use to fold the fabric into a truck. By the time the last of the fabric from the supply mandrel has passed through the plaiter, the entire batch will be loaded in the truck. The tail end is finally given a short tack to the adjacent layer at both selvages.

The plaiter has a modification which permits two of the rolls to be removed for quick release of the material. The fabric supply stand, water trough and expander rolls are mounted on a frame, which also permits sideways removal of the loop. The plaiter is then ready for unloading of the previous batch from the Barotor. When this is completed the material in the truck can be entered between the top rolls of the plaiter in preparation for the next loading.

LOADING——The rear support wheels of the rotor are momentarily hoisted off the tracks and a portion of the fabric loop passed over the rear rotor head, pulled under the wheels and placed in open width around the rotor. The inner rotor bars are then entered in the normal manner while the plaiter rolls feed the multiple layer materials from the truck. (See Figure 3). Just before the last of the bars are put in place, the fabric loop is removed from the feed rolls. Then with the addition of the last few bars to the rotor, the final portion of the fabric loop takes its place in the usual Barotor formation. No further sewing is required and the machine is ready for closing and start of the processing cycle.

UNLOADING——Unloading is commenced by removing a few bars by hand from the rotor, as is the present practice. (See Figure 4). The slack material thus released is put back into its former path through the plaiter. The plaiter rolls are then used to withdraw the fabric from the rotor and discharge it to either a truck or a scray, this operation bringing about the removal of the inner bars in the established manner. When the last of the inner bars has been withdrawn and placed back on the rack, the rear support wheels are lifted again and the loop carried back over the rotor head.

FABRIC SEPARATION——the final operation can be carried out after the next batch is loaded on the rotor from

a truck. (See Figure 5). The material is entered in the plaiter and the sewing removed from the lead end of the fabric which is then attached to a windup. As the material runs over a path similar to the preparation step, the entire batch is wound on a shell in a single thickness continuous length. If desired, this last step can be arranged to pass the material through an extractor and continuous dryer before winding. Those operations illustrated in figures 2 and 5 have been decsribed as carried out adjacent to the Barotor, but as previously pointed out, they can be performed in separate mill areas. The maximum speed for each stage has not yet been determined and will, undoubtedly, be found to vary with the material handled. In general, the first and last steps should be carried out at the maximum speed of the plaiter. Steps (2) and (3) are usually performed at lower linear speeds, but the material at this point is in multiple thickness so that the time is less than for the other two operations. Speeds in the range of 15 to 25 yards per minute are normal, which means that a 1000-yard batch of ten fabric layers is handled at rates of 150 to 250 yards per minute.

The installation illustrated in Figure 1 employs an alternate arrangement of the components. Location of the fabric supply outside the loop, eliminates the need for removal of the material from the water trough and expander rolls. In step (4), however, the fabric must be unwound from the inside of the loop when unloaded directly from the rotor to the scray without use of trucks. This necessitates a right angle turn if the fabric is to be fed directly to an extractor and dryer.

AUTOMATIC SLACK MEASUREMENT———The arrangement of feed rolls, as shown in Figures 3 and 4, has been made to agree with the present relation between fabric supply and rotor. On new installations, the arrangement shown in Figure 6 offers additional advantage. As the rolls are placed above the point of inner bar entrance, the weight of the wet fabric is made to automatically feed into the desired loop within the rotor. The drive of the rolls can also be designed to pay out a controlled amount of fabric for each jog of the rotor, thereby providing positive slack allowance for each loop. This eliminates the need for manual gauging of slack. With the improved design the operators hold the fabric against one of the outer bars while the foot pedal is actuated to jog the rotor 1/64 revolution and feed out the amount of fabric required for one loop. The feed then comes to rest and the operators hold the fabric against the next outer bar for repetition of the cycle. Faster and more uniform loading is thereby accomplished.

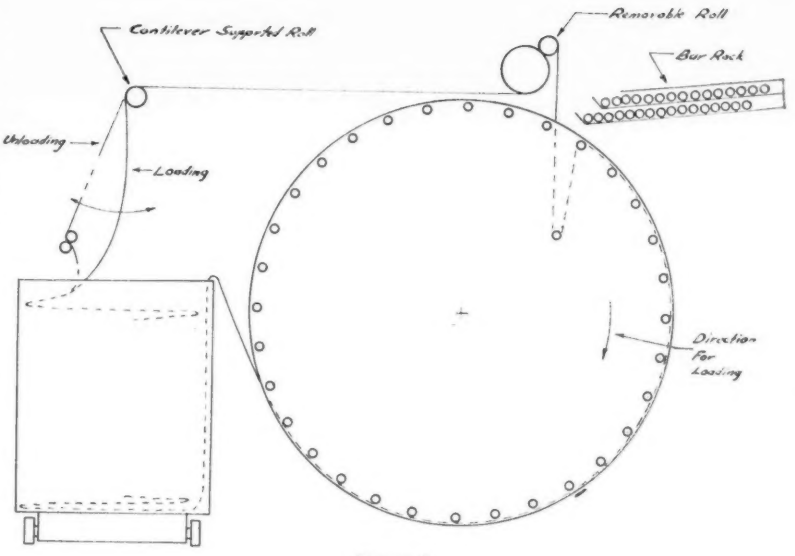


Figure 6

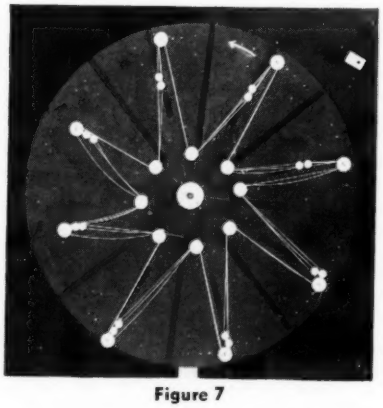


Figure 7

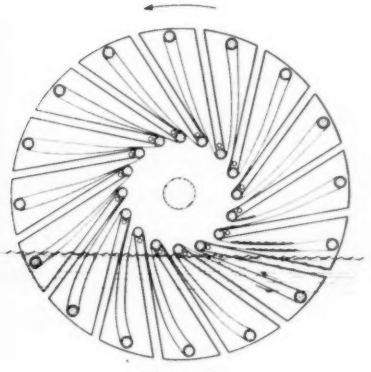


Figure 8

The combination of a gear head motor, a one revolution clutch and a variable speed drive provides a convenient arrangement for driving the rolls. The clutch is located at the input of the variable drive, which can be adjusted within the full range of slack so that each actuation of the clutch pays out an exact amount of fabric corresponding to one revolution of the input shaft.

CONCLUSION

SIMPLIFIED ROTOR———Studies by Du Pont since the Barotor was first introduced have led to two major changes, which have not heretofore been described in detail, in addition to the loading improvements. Figure 7 shows the schematic design of the original unit, and Figure 8, the new arrangement. These illustrations show eight and sixteen sets of fabric supporting bars, respectively, instead of the 64 used in the commercial machines.

The first marked change is the elimination of the spacer bars, which were formerly employed to separate the fabric layers for dyebath access. Experience has shown that dye liquor will readily pass through many unseparated layers of material by means of the paddle-like rotor action. This has resulted in the elimination of 576 bars for a 1000-yard batch.

The second important improvement is the elimination of bar movement during operation, a change that has reduced fabrication and maintenance costs. The outer bars in the original design rolled back and forth in radial slots during each turn to advance the fabric around the rotor. Study has shown that the unit can be designed so that the weight of liquid picked up by the fabric during rotation can be made to give a more effective advance without any movement of the bars. This has been facilitated by an alteration of the radial pattern of the fabric loops on the rotor to a pattern that is skewed in the direction of rotation. The fabric loops now leave the bath parallel to the surface and enter the bath at a gliding angle. This provides maximum advance at exit and minimum disturbance at entrance.

(Concluded on page 735)

HISTORICAL NOTES ON THE WET-PROCESSING INDUSTRY

VI———The Dual Life of Edward Bancroft

SIDNEY M EDELSTEIN
Dexter Chemical Corporation

This is the sixth in a series of historical articles which the textile chemist and colorist will find of interest.

The author, a prominent science historian, is the founder of an outstanding collection of great works, documents and letters in the history of chemistry and related sciences.

Articles and illustrations in this series are from the author's library.

TWO summers ago while preparing a paper on the history of static electricity I reread Van Doren's great biography of Benjamin Franklin. On many pages of this book the name of Edward Bancroft kept cropping up. This Edward Bancroft was a friend of Franklin and a spy of the worst sort. During this same summer I also had occasion to do some research in the history of 18th century dyeing. Again the name of Edward Bancroft came up, but this time as a dyer, the discoverer of the dyestuff quercitron and the author of the famous "Philosophy of Permanent Colors."

Recently I acquired for my library a scarce item having to do with Edward Bancroft's patent rights on quercitron. This led to the question . . . "was the Edward Bancroft in Franklin's biography the same person as Bancroft the dyer?" A little research revealed that it was the same man. My confusion had really been caused by the varying viewpoints of the particular authors writing about Bancroft. Where the writer was a chemist or dyer, only Bancroft's technical work was considered; where the author was an American historian, only the political side of Bancroft's life was noted; and finally, where the author was an English historian, no notice was taken of the spying or double dealing of Bancroft against the American colonies.

Let's take, for example, the Dictionary of National Biography—the British viewpoint—and compare it with the Dictionary of American Biography, the corresponding work giving the American viewpoint. In the former the first sentence of the entry under Bancroft is as follows:

"Bancroft, Edward, MD, FRS, (1744-1821): naturalist and chemist, a man of versatile talents and friend of Franklin and Priestly, published in 1769 an able tractate in defense of the liberties of the American colonies." (See Figure 1).

The first sentence of the entry in the American work is as follows:

"Bancroft, Edward (January 9, 1744-September 8, 1821): writer and inventor, better known for his double dealing and dishonorable career, was born in Westfield, Mass."

Both works continue in their own biased vein as indicated in their opening sentences.

Further study shows that there existed many errors concerning Bancroft's life and work and also many great gaps in our knowledge of this very unusual man.

Bancroft was born on Jan 9, 1744, in Westfield, Mass. He had no formal education but in later years, through his own efforts, he became well-educated in science, literature and medicine. His early life, which was quite romantic, was spent as a sailor and as a settler in Dutch Guiana. While in Dutch Guiana, even though comparatively uneducated and quite young, he made valuable studies concerning the life and religion of the natives and the natural history of the country. The result of this work was published in 1769 under the title "Essay on the Natural History of Guiana." Bancroft was only 25 years old at the time.

Just prior to 1769 Bancroft took up residence in England and in some manner became friendly with Benjamin Franklin and Joseph Priestly. Probably through their influence he pursued his scientific studies and was elected to the Royal Society and to the College of Physicians.

Sometime before the year 1771 Bancroft discovered, or at least realized the possibilities of using, the inner bark of the American Black Oak (Quercus Velutina) as a commercial dyestuff. In 1771 the British Parliament passed an act permitting the importation of quercitron (Bancroft's name for the black oak bark). On October 3, 1775, Bancroft was granted a patent, or monopoly, for the exclusive sale and importation of quercitron; and in 1785 this patent was extended until the year 1799 because of the great contribution of Bancroft's discovery to the British dyeing industry, and also because of the fact that the War with the Colonies had prevented Bancroft from making any real use out of his initial grant. (See Figure 2).

The outset of the American Revolution actually took place at about the same time as the issuance of the first patent for quercitron, and this was also the time that Bancroft began his career as a spy. While Franklin was in England, just before the war began, Bancroft acted as his special spy against the British. Within a short time, however, Bancroft was in the pay of the British and he continued to work for both sides until the end of the Revolution.

When Silas Dean went to France as an American commissioner, Franklin sent him a letter of introduction to Bancroft and both men became close friends. Bancroft helped the commissioners in their work and received pay from the American government through Dean up to the year 1783. Apparently neither Franklin nor Dean suspected the duplicity of Bancroft

REMARKS
ON THE
REVIEW of the CONTROVERSY
BETWEEN
GREAT BRITAIN and her COLONIES.
IN WHICH
The ERRORS of its AUTHOR are expoſed,
AND
The CLAIMS of the COLONIES vindicated,
Upon the EVIDENCE of
Hiſtorical FACTS and authentic RECORDS.

To which is ſubjoined,
A PROPOSAL for terminating the preſent unhappy
DISPUTE with the COLONIES;
Recovering their COMMERCE;
Reconciliating their AFFECTION;
Securing their RIGHTS;
And eſtabliſhing their DEPENDENCE on a juſt
and permanent BASIS.
Humbly ſubmitted to the Conſideration of the
BRITISH LEGISLATURE.

Conſilia qui dant prava cautis hominibus,
Et perdunt operam et deridentur turpiter.
PHOEDR. Fab. xxv.

LONDON,
Printed for T. BECKET and P. A. DE HONDT,
in the Strand.
MDCCLXIX.
Ş.

Figure 1
Title page of Bancroft's tract in defense of American liberty

(987)

ANNO VICESIMO QUINTO

Georgii III. Regis.

C A P. XXXVIII.

An Act for veſting in *Edward Bancroft*, Doctor in Phyſick, his Executors, Adminiſtrators, and Aſſigns, the ſole Property of his Invention or Diſcovery of the Uſe and Application of certain Vegetables for Dying, Staining, Printing and Painting certain valuable Colours, throughout that Part of His Majeſty's Kingdom of *Great Britain* called *England*, the Dominion of *Wales*, and Town of *Berwick upon Tweed*, for a limited Time.

DOROAS His moſt Excellent Majeſty King George the Third, by His Letters Patent under the Great Seal of Great Britain, bearing Date at Weſtminſter, the Twenty third Day of October, in the Fifteenth Year of His Reign, did give and grant unto Edward Bancroft, of Downing-ſtreet, Weſtminſter, Doctor in Phyſick, and Fellow of the Royal Society,

Figure 2
First page of the official document extending Bancroft's patent

but the other commissioners at times were suspicious. Bancroft even became the confidant of John Paul Jones and was especially commended in Dean's "Narrative to Congress" for "his help to America."

Beginning sometime in 1776 Bancroft carried on a similar career as a British spy. In December, 1776, he arranged to supply information to the British for the annual sum of 400 pounds and this was later increased to 1000 pounds. In order to make his position appear even more plausible to the Americans, the British arranged to have him arrested in 1777 for corresponding with Dean and then released him.

Bancroft apparently enjoyed his "cloak-and-dagger" work. He sometimes used the name of Edwards and among his special methods for transmitting information was the placing of correspondence in a bottle, which he concealed in the trunk of a tree near the Tulieres Garden in Paris. Bancroft revealed all of Dean's dealings with the French and furnished the British with abstracts of American treaties.

Bancroft seemed always to be hungry for money and was a natural speculator. Sometimes he suffered substantial losses, however. He even speculated in England on the advance news of Burgoyne's defeat which he was able to get from the Americans. Bancroft was especially commended by the English officials for his work, but at times even they were suspicious. In a "memorial" to the Marquis of Carmarthen, Bancroft especially claimed credit for furnishing information about the movement of supplies, French and American ships, and particularly, of the sailing of D'Estang's fleet. The authoritative British Dictionary of National Biography makes no mention whatsoever of this side of Bancroft's life.

After the Revolution, Bancroft renewed his friendships with Franklin and other Americans and made several trips to North and South America. Apparently he received a substantial income from the importation of quercitron into Britain. In addition he continued his studies in the field of dyeing, and in 1794 published "The Experimental Researches Concerning the Philosophy of Permanent Colors."

In 1799 Bancroft's patent was due to expire and he petitioned Parliament for an additional seven-year extension. In his petition he agreed to make, as a part of the extension, an agreement to furnish quercitron to dyers at the same low price which he previously had established notwithstanding the fact that most imported dyewoods had trebled in price. While Bancroft had the support of a majority of the dyers of the southern part of Britain, and while the petition did pass the House of Commons, it was lost in the House of Lords through the efforts of certain dyers from the northern part, who argued that the removal of Bancroft's monopoly would reduce the cost of the dye. Peculiarly enough, after the expiration of Bancroft's patent in 1799, the price of the bark rose to three times that which Bancroft had agreed to furnish it. In Bancroft's words:

Figure 3
American Black Oak, source of quercitron

EXPERIMENTAL RESEARCHES

CONCERNING THE

PHILOSOPHY

OF

PERMANENT COLOURS;

AND THE

BEST MEANS OF PRODUCING THEM,

BY

DYEING, CALICO PRINTING, &c.

BY

EDWARD BANCROFT, M. D.

FELLOW OF THE ROYAL SOCIETY OF LONDON, AND OF THE AMERICAN ACADEMY OF ARTS AND SCIENCES, OF THE STATE OF MASSACHUSETTS BAY.

" Cet art (de la teinture) est un des plus utiles et des plus merveil-
" leux qu'on connoisse ; & si *quelque un peut inspirer un noble orgueil à*
" *l'homme, c'est celui là:* non seulment il a procuré le moyen de suivre
" et d'imiter la nature dans la richesse & l'eclat des couleurs; mais
" il paroit l'avoir surpassé en donnant plus d'eclat, plus de fixité & plus
" de solidité aux couleurs fugaces & passageres dont elle a revetu
" tous les corps qui composent ce globe."

CHAPTAL, *Elemens de Chimie*, tom. iii. p. 185.

VOL. I.

London:

PRINTED FOR T. CADELL AND W. DAVIES, IN THE STRAND,

By G. SIDNEY, Northumberland-street.

1813.

Figure 4
Title page of Volume I in enlarged edition of "Permanent Colours"

"This is the only instance, I believe, in which an invention ever became more costly after the expiration of a monopoly granted to remunerate the inventor than it was during the continuance thereof, and it has demonstrated most incontrovertibly that my opponents were greatly deceived, and I was greatly wronged."

Little is known about the details of Bancroft's life from this time until his death at Margate on Sept 8, 1821. Apparently he lived under reduced circumstances and devoted a considerable part of his time to the study of dyeing and chemistry. In 1813 he republished his "Experimental Researches Concerning the Philosophy of Permanent Colors" in a greatly enlarged and improved form.

Bancroft unquestionably made two great contributions to the dyeing industry.

First, his discovery of the use of the quercitron bark not only furnished an important article of trade between America and Europe for a period of more than 100 years, but it saved the dyers of the world many millions of dollars in their dyeing costs. Quercitron is the inner bark of the black oak, which is known today as quercus velutina, a native tree of the Middle and Southern United States. (See Figuré 3). The color imparted by this dyestuff is yellow; hence the name quercitron, meaning oak yellow. This name

(Concluded on page 735)

• PATENT DIGEST •

PAUL WENGRAF

SHRINKPROOFING WOOL ———Acid Salts of Deacetylated Chitin A, 2, 04

U S Pat 2,669,529
(Montclair Research Corp———Ellis Foster Co———Rust——Feb 16, 1954)

Among the older methods for shrinkproofing wool are those comprising chlorinating and other oxidative processes, and in recent times, various treatments with thermoplastic resin polymers or copolymers or with thermosetting condensates (e g, melamine) have been advocated. Disadvantages, such as possible fiber damage and reduced wear resistance in the case of the chlorine treatments, and insufficient stability of the solutions and/or the final effect, etc, in the case of resin treatments, are discussed in the preamble to the present specification.

The inventor recommends the use of water-soluble acid salts of *undegraded, at least partially deacetylated, chitin* as a shrinkproofing agent. Chitin itself is known as a polymeric acetamino carbohydrate, which may be found in the shells of crabs, lobsters or other crustaceae, and also in "exo-skeletons" of some insects. The building unit of chitin is acetylated chitosamine. This product may be partially deacetylated without any degradation, i e, without depolymerization by methods available in the art (and probably, according to some Du Pont patents quoted below, by controlled alkali treatment). The acetyl groups are removed to a great extent but not entirely. The best results are obtained when the viscosity of a solution of 0.25% deacetylated chitin in 0.25% acetic acid at 25° C measures 5-20 centipoises. In this event 70-90% *free* aminogroups are present. The relative viscosity is 2.5-50 times that of glycerine at 27.5° C.

Acid salts of the deacetylated product are soluble in water to a great extent. Acetic acid is preferred, but other monobasic aliphatic acids, higher fatty acids, or aromatic or inorganic acids can be used as well.

2-5% of the product deposited in the fiber will be sufficient for a good shrinkproofing effect. The goods are impregnated and dried at relatively low temperatures, then baked at 100° C or more. The effect is logically correlated to time and baking temperature. 5-20 minutes at 130° C is said to give a very durable, satisfactory result. Polyalcohols and polyglycols (Carbowaxes) having plasticizing effects, or other ingredients, such as sorbitol combined with hexamethylene tetramine or allylsucrose, may be added. Modifying deacetylated chitin by organic halides is suggested for increasing wear resistance and flexibility of the deposited film. Faster curing, i e, insolubilizing of the compound, is attained by condensing deacetylated chitin with allylbromide, which causes allylation according to the equation

$$C_6H_9O_4NH_2 + BrCH_2-CH=CH_2 \xrightarrow[NaOH]{} C_6H_9O_4N(H)-CH_2CH=CH_2 + NaBr + H_2O$$

or possibly also etherification on a free hydroxygroup. While insolubilization of the chitin generally is effected by heat (baking), a *chemical setting process* by surface-active anionic agents, e g, organic sulfates, phosphates or the like (claim 5), or by salts (claim 4), is also within the scope of this invention.

Example: Samples of wool flannel are immersed in the padding solution, which contains 2.5% solids, prepared by dissolving 2.5 p of deacetylated chitin in 97.5 p of a solution of 2.5 p glacial acetic acid in 95 p water. The samples are then dried at 60° and cured at 130° C for 5 minutes (or at 120° C for 20 minutes, at 110° C for 30 minutes or at 100° C for 45 minutes). The resultant area shrinkage is found to be 0-0.5% as compared with 26.5% for the nontreated material.

Other examples refer to the effect of other additives and to results obtained using some anion-active surfactants as setting agents.

References cited by the Patent Office:

U S Pat 2,173,243 (U S Rubber Company/1939): treating fibrous material for sizing, finishing, softening, etc, with dispersions of pigments, resins, or softeners adjusted to the isoelectric pH value and protected from coagulation by any known protective colloid. The dispersion is adsorbed to a maximum under these conditions upon contact with textiles and thereafter coagulated.

U S Pat 2,047,217 (Du Pont/1936) describes quite generally the use of salts of deacetylated chitin, especially of the acetate, as emulsifying agents for water-repellent substances (waxes, rosins, rubber and the like) in treating paper or textiles.

Further detailed information on the technical use of deacetylated chitin may be found in the series of British Patents 458,813-16 and 458,839 assigned to Du Pont. Brit P 458,815 particularly points out the use of this product for fixing water repellents. Shrinkproofing finishes are not mentioned in any of these references.

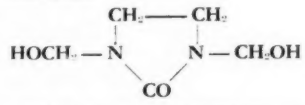
DURABLE STARCH FINISHES ———Adding Imidazolidone Derivatives G, 2, 01

U S Pat 2,661,312
(Du Pont———Richardson———Dec 1, 1953)

This specification mentions earlier references, which describe compositions of starch pastes combined with resin precondensates. In using these, premature condensation and undesirable thickening of the paste can be avoided only by working at very low temperatures. On the other hand, impregnation with warm finishing pastes is preferred in practice in order to effect better impregnation. Furthermore it is not economical to cool down the starch paste before adding the resin precondensate.

It has been found that 1-3-bis (hydroxymethyl)-2-imidazolidone, used as a specific resin condensate, can stand temperatures up to the boiling point for several hours. Curing, i e, insolubilizing of the resin, occurs only at temperatures above the boiling point.

The aforementioned precondensate is prepared, according to U S Pat 2,373,136 (see references below), by reacting ethylene urea with formaldehyde giving the compound



This differs from dimethylolurea in the ring formation through the ethylene group, possibly accounting for the higher stability of this product.

Example: a pearl corn starch paste is dissolved 1:2 in water at room temperature. Equal parts of this starch solution and a 50% solution of the precondensate described above, plus a small amount of tartaric acid as a catalyst, are added to boiling water. This solution is held for about 30 minutes at 165-170° F without its undergoing any change. It is used at this temperature for padding a cotton sheet, which is squeezed, dried and baked at 350° F. After the neutralizing and pressing operations, the fabric is found to have improved stiffness, body, crease resistance and dimensional stability.

References cited by the Patent Office:

U S Pat 2,373,136 (Du Pont/1945) refers to the preparation of ethylene urea derivatives as mentioned above. These compounds quite generally are recommended as resins for creaseproofing, stabilizing, etc.

U S Pat 2,238,839 (Watkins/1941) describes "re-inforced" creaseproof finishes made from aldehyde-urea or aldehyde-thiourea condensates combined with customary filling agents, such as casein, albumen or starch preparations.

U S Pat 2,121,006 (Raduner-Bener/1938): stabilizing textiles against srinkage by applying artificial resin solutions (urea or thiourea-formaldehyde) optionally combined with starch finishes or the like.

Proceedings of the

American Association of Textile Chemists and Colorists

Copyright, 1954, American Association of Textile Chemists and Colorists

Editor of Proceedings: Carl Z Draves, 30 Lahey St, New Hyde Park, N Y

OCTOBER 25, 1954

AATCC CALENDAR

(A general calendar of events may be found on page 740)

COUNCIL
1954—Nov 19 (Hotel Roosevelt, New York).
1955—Jan 21 (Hotel Statler, New York), Apr 15, June 17.

NATIONAL CONVENTIONS
Sept 22-25, 1955 (Chalfonte-Haddon Hall, Atlantic City, N J; 1956 (Waldorf-Astoria, New York); 1957 (Boston).

HUDSON-MOHAWK SECTION
Oct 29 (Johnstown, N Y); Dec 2, Jan 14, Feb 25, April 15 (Albany, N Y); May 20 (Ladies Night); June 24 (Annual Outing).

MID-WEST SECTION
Oct 30 (Bismarck Hotel, Chicago, Ill).

NEW YORK SECTION
November 19 (Kohler's Swiss Chalet, Rochelle Park, N J); January 28 (Georgian Room, Hotel Statler, New York); February 25 (Skytop, Hotel Statler, New York); March 18, April 22, May 20 (Kohler's Swiss Chalet, Rochelle Park, N J).

NORTHERN NEW ENGLAND SECTION
Dec 10 (Woodland Golf Club, Auburndale, Mass).

PACIFIC SOUTHWEST SECTION
Oct 30 (Outing—Ojai, Calif); Dec 3 (Gourmet Restaurant, Beverly Hills, Calif).

PHILADELPHIA SECTION
Oct 29 (Penn-Sherwood Hotel, Philadelphia, Pa); Dec 3 (Kugler's Restaurant, Philadelphia).

RHODE ISLAND SECTION
Dec 2 (Annual Meeting—Sheraton Biltmore, Providence, R I).

SOUTH CENTRAL SECTION
Dec 4 (Hotel Patten, Chattanooga).

SOUTHEASTERN SECTION
Dec 11 (Atlanta-Biltmore, Atlanta, Ga).

WASHINGTON SECTION
Dec 8 (Pepco Auditorium, Washington); Feb 11 (Washington); May 20.

WESTERN NEW ENGLAND SECTION
Nov 12, Dec 10 (Rapp's Restaurant, Shelton, Conn).

WESTERN NEW YORK SECTION
Nov 27 (Hamilton, Ont); Jan 14 (Buffalo); Mar 4 (Hamilton, Ont); April 22 (Buffalo); June 24 (Outing).

ACTIVITIES OF THE LOCAL SECTIONS

Mid-West Section Establishes Annual Scholarship Award

SONS and daughters of members of the Mid-West Section are now eligible for an Annual Scholarship Award, which has been established by the Section.

The total amount of the award, which will be for one year, will not exceed four hundred dollars. The recipient will be selected each Spring by a committee of Mid-West Section members appointed by the Chairman.

Requirements on which the Committee will base their selection are as follows:

a) Each candidate must satisfy the Committee that he or she is enrolled in, or will apply for admission to the textile institute, school or college of his or her choice.

b) Academic standing in the first three and one-half years of secondary school, or the period of enrollment in college.

c) Extra-curricular activities.

d) Financial need.

—♦ ♦—

Washington

FUTURE meetings of the Washington Section will feature the following:

Dec 8 — Pepco Auditorium — "Creative Directions in Color Photography," Ralph M Evans, Color Technology Div, Eastman Kodak Co. (This meeting will be held in conjunction with other member-bodies of the Inter-Society Color Council.)

Feb 11 — "Mechanism of Wrinkle-Resistant Finishes for Cellulosic Fabrics," Theodore F Cooke, American Cyanamid Co.

May 20 — Symposium on "The Role of Government in the Textile Industry." Panelists will include William D Appel, National Bureau of Standards; G E Hilbert, U S Dept of Agriculture; Harvey Hannah, Federal Trade Commission; and Henry Thurston, U S Dept of Commerce.

Western New England

SOCRATES V VANIOTIS, American Cyanamid Co, was elected to head the Western New England Section for the coming year at a regular meeting on Friday, October 1, 1954, at Rapp's Restaurant, Shelton, Conn.

Also elected were John E Hirn, Sr, *vice chairman;* Nathaniel J Glade, *secretary;* Thomas J Gillick, Jr, *treasurer;* and Rudolph C Geering, *councilor.* The following were elected to the Sectional Committee: A N Demarco, A W Goodwin, A H Rant, and P W Reeves.

Approximately 70 members and guests were on hand to hear Irvin H Welch, Jr, Textile Research Dept, American Viscose Corp, discuss "Washable Rayons."

—♦ ♦—

Philadelphia

APPROXIMATELY 150 members and guests of the Philadelphia Section attended the first meeting of the 1954-55 season on Sept 24 at the Penn-Sherwood Hotel, with chairman F V Traut presiding.

Speakers of the evening were A Frank Tesi, Celanese Corp of America, on the subject "Color Performance Possibilities of Solution-Dyed Acetate," and L Leslie Walmsley, American Viscose Corp, on "Spun-Dyed Viscose."

—♦ ♦—

New York

A MEETING of the New York Section was held on Friday evening, October 15th, 1954 at Kohler's Swiss Chalet, Rochelle Park, New Jersey. Due to lack of electricity and the presence of hurricane weather, the presentation of the paper by Francis S Richardson on the Barotor in practical application was postponed.

The following nominating committee was appointed to select nominees for councilors for election at the November meeting: Henry L Young, *chairman,* Weldon G Helmus and Francis S Richardson.

*AATCC Research Newsletter No. 17

NEWS IN GENERAL

On the occasion of meetings of ASTM committee D-13 and other technical groups in Washington, D C on October 18 and 19, AATCC again clarified its position as regards the development of end-use performance specifications by various industry groups in which its test methods are called for. In a joint statement of officials attending the meetings, it was made clear "that AATCC always has been, and still is, concerned directly with standard test methods, and that it does not concern itself with standard performance specifications; that AATCC does co-operate with other societies, but members participating in such activities do so only as technical advisors on standard test methods."

At a meeting of the Steering Committee of the Industry Advisory Committee on Commercial Standard CS 59-44, it was voted that this standard is out-dated and in need of revision. The committee also moved to proceed with developing proposals for revisions for later action of the whole committee.

Good progress is being made by the Joint Committee of ASTM and AATCC, which also met in Washington on October 19, for its sixth meeting. The committee is seeking ultimately to align the textile tests of each organization both in scope and procedure.

Messrs Appel and Little have completed a review of the proposed international colorfastness tests approved at the ISO meetings in Scarborough, England last June. Pending completion of a similar review by the Society of Dyers & Colourists, Arthur F B Nall of the British Standards Institution will prepare a final draft for circulation among the technical groups in the several co-operating countries. Only upon their unanimous approval of the proposed standards can they be adopted by the International Standards Organization as international standards.

In a new edition of its popular booklet "Dyes and Their Properties," the Franklin Process Company will reproduce for the first time the complete AATCC Standard Test Method 36-54, Colorfastness of Cotton, Linen and Manufactured Organic Fibers to Commercial Laundering and Domestic Washing. AATCC has always encouraged the publication of its tests in industry-produced educational material as beneficial to the public relations interests of the textile industry.

AT RESEARCH HEADQUARTERS

A suggestion has been made that AATCC Research Newsletters be mailed separately to interested persons in corporate member firms. The letters will continue to be published in ADR, but for the convenience of those who wish separate copies AATCC is considering a separate mailing. Let us know if you would want to be on this special mailing list. Write to Dick Frey at headquarters.

Miss Jean Dressel of Sears, Roebuck & Co, Chicago Laboratories technical staff, spent a week at headquarters recently studying the Accelorotor and its applications to consumer textile problems. The tremendous savings in time possible with this device in many types of end-use evaluations has attracted considerable interest among laboratories that handle large volumes of testing.

Testfabrics, Inc reports the development of a new type super-multifiber test cloth composed of eleven different fibers in spun form arranged in alphabetical order in quarter-inch warp stripes. The cloth was designed as an aid to determining how particular dyes exhaust onto the different fibers currently in production. Designed primarily for commercial requirements,the new cloth is not specified in any AATCC tests and thus is not included with the AATCC-controlled test fabrics produced by this firm.

COMMITTEE NEWS

The following chairmen will have reached the end of their three-year terms of office next January 1st, necessitating new appointments to succeed them: L L Heffner, Evaluation of Fire-Resistant Textiles; T E Bell, Bleaching; G Wiseman, Standard Soils; A E Johnson, Drycleaning Test Methods; C R Bellwood, Water Resistance of Fabrics; C J Monego, Visual Aids for Evaluating Color Differences; and W H Pardey, Colorfastness to Fulling. A new chairman will also be named to the Color Committee to fill the vacancy created by the death of I H Godlove. The outgoing chairmen have been contacted by TCR chairman Chas W Dorn for recommendations on their successors.

* Published every other issue of the AMERICAN DYESTUFF REPORTER.

EMPLOYMENT REGISTER

54-35

Education: BS, textile institute, textile engg.

Experience: asst designer.

Age: 26; single; veteran; location not restricted.

10-25, 11-8

54-36

Education: MS, textile institute, textile chemistry & dyeing.

Experience: chemist and laboratory manager.

Age: 25; married; veteran; New York or New Jersey preferred but not essential.

10-25, 11-8

54-37

Education: BS, Mass Inst Tech, chemistry.

Experience: chemist with company manufacturing woolens and worsteds since 1921.

Age: 59; married; references; New England preferred but not essential.

10-25, 11-8

SECRETARY'S FINANCIAL REPORT—JULY 31, 1954

	Applications	*Dues, Regular & Reinstate*	*Dues, Corporate & Sustaining*	*Miscellaneous*	*Totals*
Received & transmitted to Treasurer August 1, 1953 to May 20, 1954	3982.00	45440.25	47142.50	26677.66	123242.41
May 20 to July 31, 1954	401.00	251.00	425.00	2942.81*	4019.81
Total in fiscal year ending Ju'y 31, 1954	4383.00	45691.25	47567.50	29620.47	127262.22
Leaving deposited by Treasurer with Secretary,					3176.93

* Miscellaneous Items,

Reporters and reprints	86.13
Year Books,	270.70
Crockmeters,	450.45
Crock cloth,	177.71
Multifiber cloth,	390.16
Moth cloth	49.38
Water test apparatus,	34.02
Dyed standards,	39.00
Knitted tubing	10.00
Gas-fading units	23.00
Buttons	4.50
Color transfer charts	132.50
Colour Index, second instal'ment payments	62.00
Analytical Methods,	106.00
Application of Vat Dyes,	346.55
Cotton skeins	51.35
Standard grease wool,	28.50
Launder-Ometer royalties,	145.00
Accelerotor liners,	330.50
American Viscose Scholarship,	154.78
Return of travel expense funds,	37.08
Grey scales	13.50
	2942.81

H C CHAPIN, *secretary*

"A Century of Chemical Progress"

THEME SELECTED FOR PERKIN CENTENNIAL

THE theme, "A Century of Chemical Progress," has been selected for the Perkin Centennial, which will be held at the Waldorf Astoria Hotel, New York, N Y, during the week of September 10, 1956, according to an announcement by Raymond W Jacoby, chairman of the Executive Committee for the event.

An expansion of the theme follows:

"In 1856, a lad of 18, William H Perkin, was experimenting in his home laboratory in an effort to produce synthetic quinine. At the completion of one of his trials, he found a violet-colored material and, although this effort to produce a synthetic quinine failed, he had discovered the first synthetic dye—mauve. With the desire for color being a natural human urge, he realized the value of his discovery. Further research soon produced other dyes.

"From coal, water, air, petroleum and natural gas, numerous organic chemicals were produced. These were compounded and reacted and the number of new dyes increased rapidly until, today, we have over three thousand synthetic dyes, capable of producing all the hues of the rainbow. Truly, mauve was the key to the rainbow.

"The restless and inquisitive spirit of man led to the realization that these organic chemical compounds had many valuable possibilities other than the production of dyes. They became the source of many wonder pharmaceuticals, so that, oddly enough, an effort to produce a drug which was valuable in its day, subsequently became the starting point for the production of drugs with properties undreamed of years ago.

"The growth and expansion of the synthetic organic chemical industry proceeded at a rapidly accelerated rate. Insecticides, fungicides, soil conditioners and fertilizers improved the quality and quantity of our food supply. The development continued until it affected all phases of life.

"From this beginning, the ever broadening field covered synthetic rubber, wood preservatives, lacquers which both protect and beautify, cosmetics, new fibers to impart qualities to fabrics which previously were non-existent, plastics which serve in manifold ways, hormones, vitamins, mildew inhibitors, water repellents, solvents, detergents, and countless other items which have brought us into a new era.

"Through these developments, the span of human life has been lengthened and that span has been made more enjoyable, richer and fuller with many blessings."

AUDITOR'S REPORT

TO the Members of the
American Association of Textile Chemists and Colorists:

We have made an examination of the accompanying statement and supporting schedule summarizing the receipts and disbursements of the treasurer and secretary of the American Association of Textile Chemists and Colorists for the year ending July 31, 1954. In connection therewith, we examined or tested the cashbooks and other records supporting the receipts and disbursements by methods and to the extent we deemed appropriate, but we did not communicate with the members to confirm the amount of recorded receipts from dues and subscriptions or from advance subscriptions for the *Colour Index*.

In our opinion, the accompanying statement and supporting schedule fairly summarize the recorded receipts and disbursements (excluding receipts and disbursements on sales and purchases of investment securities) of the Association for the year ending July 31, 1954 and the balances of cash in banks, advance expense funds, and securities owned as of that date.

PRICE WATERHOUSE & CO.

75 Federal Street
Boston 10, Mass.
August 18, 1954

AMERICAN ASSOCIATION OF TEXTILE CHEMISTS AND COLORISTS
SCHEDULE OF MISCELLANEOUS INCOME
FOR THE YEAR ENDING JULY 31, 1954

Research:		
Launder-Ometer royalties	$ 285.00	
Grey scales	108.50	
Accelerotor liners	486.10	$ 879.60
Sales of technical supplies, royalties, etc.:		
Yearbooks	$2,520.57	
Dye standards	447.00	
Knitted tubing	293.60	
Dyestuff Reporters and reprints	422.48	
Crock-meter cloth	665.95	
Crock meters	3,110.43	
Moth cloth	244.93	
Multi-fibre cloth royalties	1,877.87	
Flammability tester royalties	4.00	
Water-test apparatus	221.17	
Gas fading units	205.00	
Color transference charts	824.36	
Analytical methods	687.15	
Buttons	25.50	
Application of Vat Dyes Monograph	3,773.37	
Cotton skeins commissions	226.29	
Standard grease wool	216.00	
Apparatus catalogs and dry cleaning kits	6.10	
Accelerotor royalties	144.90	15,916.67
Total miscellaneous income		$16,796.27

TREASURER'S REPORT

AMERICAN ASSOCIATION OF TEXTILE CHEMISTS AND COLORISTS
STATEMENT SUMMARIZING RECEIPTS AND DISBURSEMENTS FOR THE YEAR ENDING JULY 31, 1954
(excluding receipts and disbursements on sales and purchases of investment securities)

		Total	Allocation of Funds: Special Research Projects: General Research	Philadelphia Section Research Project	Colour Index—Second Edition (See Footnote): Advance Subscriptions	Colour Index—Second Edition (See Footnote): Disbursements	American Dyestuff Reporter Prize Award Fund	American Viscose Co. Scholarship Fund	Olney Medal Fund	Convention Fund	Technical Supplies	General
Receipts:												
Membership dues:												
Corporate memberships		$ 46,845.00	$ 44,617.50									$ 2,227.50
Sustaining memberships		722.50	722.50									...
Senior, Junior, Associate and Student Memberships		50,074.25	...									50,074.25
Total membership dues		$ 97,641.75	$ 45,340.00									$ 52,301.75
American Dyestuff Reporter—Prize Award Fund		1,044.65	...				$1,044.65					...
American Viscose Company—Scholarship Fund		775.78	...					$ 775.78				...
Annual meeting registrations—net amount received from Mid-West Section		15,677.64								$15,677.64		...
Dividends and interest received (net)		9,710.38	1,818.19		$ 298.01		198.90		$ 403.35			6,991.93
Advance subscriptions to Colour Index		8,539.88			8,539.88							...
Miscellaneous income, per accompanying schedule		16,796.27	879.60								$15,916.67	...
Total receipts		$150,186.35	$ 48,037.79		$ 8,837.89		$1,243.55	$ 775.78	$ 403.35	$15,677.64	$15,916.67	$ 59,293.68
Disbursements:												
Research salaries and expenses		$ 54,858.49	$ 54,858.49									$ ...
Expenses chargeable to Colour Index Project (see footnote)		31.52	...			$ 31.52						...
Howes Publishing Company—Subscriptions to American Dyestuff Reporter		20,309.86	...								$ 2,119.31	18,190.55
Disbursements for account of Olney Medal Fund		231.18	...						$ 231.18			...
Disbursements for account of American Viscose Company Scholarship		1,324.36	...					$1,324.36				...
Secretary's expenses, clerical assistance, etc.		28,624.35	...								6,532.53	22,091.82
American Dyetuff Reporter Prize Award		200.00	...				$ 200.00					...
Publicity		2,853.16	2,229.77							$ 363.21		260.18
Purchases of technical supplies for resale		2,209.62	...								2,209.62	...
Section membership allowances		4,503.56	...							2,467.76		2,035.80
Other general expenses		851.59	...									851.59
Total disbursements		$115,997.69	$ 57,088.26			$ 31.52	$ 200.00	$1,324.36	$ 231.18	$ 2,830.97	$10,861.46	$ 43,429.94
Excess (deficiency) of receipts over disbursements		$ 34,188.66	($ 9,050.47)		$ 8,837.89	($ 31.52)	$1,043.55	($ 548.58)	$ 172.17	$12,846.67	$ 5,055.21	$ 15,863.74
Cash in banks, advance expense funds and investments (at cost), July 31, 1953		280,250.56	125,349.37	$2,000.00	11,452.00	(41,661.53)	6,155.35	548.58	6,588.51	7,761.89	12,666.34	149,390.05
Loss on sale of United Fruit Company common stock		(827.38)										(827.38)
Cash in banks, advance expense funds and investments (at cost), July 31, 1954		$313,611.84	$116,298.90	$2,000.00	$20,289.89	($41,693.05)	$7,198.90	$ ...	$6,760.68	$20,608.56	$17,721.55	$164,426.41

Summary of cash in banks, advance expense funds and investments at July 31, 1954:		
Cash in banks		$100,404.82
Advance expense funds		5,500.00
United States Government obligations, at cost:		
Savings Bonds (value at maturity $32,000; redemption value at July 31, 1954 $30,854)	$29,400.00	
Treasury Bonds, etc. (face amount $90.00; value based on market quotations July 31, 1954 $90,268)	90,064.56	119,464.56
Other marketable securities, at cost (value based on market quotations July 31, 1954 $132,611)		88,242.46
		$313,611.84

Note: Under the agreement dated January 18, 1952, between the Association and The Society of Dyers and Colourists of Bradford, England, all costs incurred in connection with the compilation, printing, distributing, etc. of the "Colour Index (Second Edition)" are to be borne equally by the parties thereto. Future net losses or revenues arising from the production and sale of this work are likewise to be borne or shared equally by the parties. The Association's total commitment under the agreement, representing half the presently estimated cost of production, is approximately $126,000. The Association and the British Society, each in their respective territories, are receiving subscriptions in advance of publication to assist in financing the project. As at July 31, 1954, the Association on its part had expended in salaries and other costs a total amount of $41,693.05 and had received in advance subscriptions a total amount of $20,289.89 (including interest of $298.01 on funds deposited in savings banks accounts). Information is not currently available as to the aggregate amount of expenditures made and advance subscriptions received by the British Society. The joint agreement requires annual equalization settlements between the parties with respect to costs paid and revenues received by each. Because of British exchange restrictions, the annual equalization settlements through December 31, 1953 have been waived by mutual agreement.

Respectfully submitted:
ALBERT E. SAMPSON
Treasurer
American Association of Textile Chemists and Colorists

A NEW METHOD OF VAT-DYEING KNIT GOODS*

ROBERT D BLUM, JR

May Hosiery Mills

In vat-dyeing seamless hosiery and other knit goods of various fibers, the chief problems encountered are penetration, levelness, reproducibility, and union problems, where multiple fibers are used in construction. A novel method has been developed and used in production in overcoming these difficulties. The method has been particularly successful for attaining levelness for pastel shades and penetration for heavy shades on tightly knit fabrics. In addition, it makes possible the utilization of vat dyestuffs heretofore not applicable by conventional dyeing methods. Several unusual applications of the method will be discussed, including a simple method of topping vat dyeings with conventional vat dyestuffs without losing any of the base vat dyestuffs of the original shade.

INTRODUCTION

THE application of vat dyes to knit goods, such as hosiery, has presented the dyer with numerous levelness and penetration problems. These problems are largely due to the rapid exhaustion rate of many vat dyestuffs and to the construction features of knitted goods where certain portions of the fabric are more tightly knit than other areas, resulting in a difference in dye absorption. The term "levelness" is construed as a broad term and will be interpreted in this paper as including both penetration and even distribution of the dyes throughout all parts of the fabric.

The exhaustion rate and the rate of diffusion are generally regarded as the critcal factors in the promotion of levelness *(1, 2, and 3)* and can be controlled, with limitations, by means of retarding agents or by the temperature-gradient method.

The practice, carried out by hosiery dyers, of using a weak reducing agent, such as a sugar, a dextrine or sodium sulfoxylateformaldehyde (hereafter referred to as "sulfoxylate" or "Discolite") for the purpose of attaining a partial or slow initial reduction, is well known. In such cases, however, sodium hydrosulfite (hereafter referred to as "hydro") is used as the primary reducing agent. The methods which will be discussed herein are based on the use of Discolite as the primary reducing agent, without hydro, and also in conjunction with small quantities of hydro in certain special applications.

The success of the methods suggested herein is based on the experimentally determined facts *(1)* that

a) Reduction potentials developed with Discolite (sulfoxylate) are comparable to those obtained with hydro at 160° F and above under similar concentrations and conditions.

b) The leucos, or reduced form, of vat dyestuffs, obtained with Discolite are different in physical and chemical properties from the hydro leucos of the same dyestuffs.

c) The reducing agent Discolite is more stable at high temperatures and at varying pH values than hydro.

* A revision of a paper presented originally at the 1953 Convention in Chicago, Ill.

EXPERIMENTAL

APPARATUS USED——In all experimental determinations of redox potentials, a portable Foxboro Dynalog Redox Recorder was used, and, in all pH determinations, a Macbeth Model A Laboratory pH meter. All dyeings were carried out at a 20-to-1 bath ratio. Laboratory dyeings were made in stainless-steel beakers, with temperature controlled by water baths. Production dyeings were made in both open-top paddle machines and Smith-Drum closed rotary machines.

REDUCTION POTENTIALS —— Reference is made to the Intersectional Contest Paper of the South Central Section *(1)*, in which comparisons of reduction potentials between sulfoxylate and hydro are given as well as comparisons of typical dyeing curves for both sulfoxylate dyeings and hydro dyeings relating redox potentials to time. It is seen that, with hydro, immediate high potentials are developed at low temperatures, and that with increased temperature, the increase in potential is developed at a moderate rate, while decreased temperature reduces the potential at a very slow rate. However, with sulfoxylate, the initial potentials developed at low temperatures are much lower than with hydro, but a rapid increase in potential occurs as temperature increases, and a moderately rapid decrease in potential occurs as temperature decreases. The observation that reduction with sulfoxylate is, to a greater degree, a function of time than of temperature or alkalinity (which are also important factors) is graphically illustrated by testing a dyebath of known reduction potential of 1,000-1,200 millivolts with Vat Yellow G test paper, which has a leuco potential of less than 800 millivolts. The paper does not begin to turn blue unless it remains in the bath for a period of at least ten seconds; whereas, in a

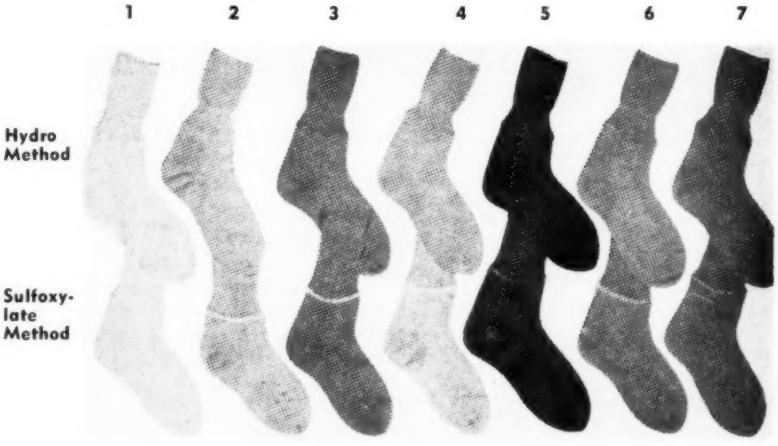


Figure 1
A comparison of the dye penetration of the sulfoxylate and hydro methods
1—Anth Vat Yellow CC Dbl, 0.5% owf
2—Indigoid Vat Pink FF Dbl, 1.5% owf
3—Anth Vat Blue-Green Y Dbl, 1.5% owf
4—Indigoid Vat Brown G, 1.5% owf
5—Ponsol Vat Navy Blue, 10.0% owf
6—Anth Vat Jade Green Dbl, 2.0% owf
7—Anth Vat Blue BF Dbl, 1.5% owf

hydro solution of lower potential, the color change is immediate.

APPLICATION TO COTTON

In the application of vat dyes to both mercerized and unmercerized cotton goods, three general methods were attempted, depending upon the dyestuffs used and the construction of the goods.

In the methods outlined below, all dyeings were made at 20/1 bath-to-goods ratio, such as would normally be used in hosiery or knit goods dyeing in paddle wheel or rotary machines or on a jig.

METHOD A

1) Scour goods in conventional manner and drop bath.

2) Refill bath and set at 130-140° F with a dispersing agent and a sequestering agent.

3) Disperse the dyestuff and add in 2-5 minutes at 130-140° F; run 10 minutes at 130-140° F.

4) Add 8-15% dissolved caustic soda and 8-15% sulfoxylate; heat to 160° F.

5) Raise temp slowly in 10-15 minutes to 190-200° F.

6) Continue dyeing at 190°F, cooling to 160-170° F, adding salt where necessary for exhaustion for 10-20 minutes.

7) Flush goods, oxidize, soap and soften in conventional manner.

METHOD B

Same as Method A, except approximately 2/3 of the quantity of sulfoxylate is used, and in the place of the remainder, hydrosulfite is added at the end of Step 6. The dyeing is run for an additional 5 minutes.

METHOD C

1) Set bath with a wetting agent, a sequestering agent, and 8-15% caustic soda, and then heat to 140° F; enter goods in bath and heat further to 200° F; run for 10 minutes at 200° F.

2) Add approximately 1/3 (2-5%) of the required sulfoxylate and run 5 minutes at 200° F.

3) Disperse the dyestuff at 150-160° F, adding the remainder of the required sulfoxylate (3-10%) to the dispersed dye until the sulfoxylate is dissolved; add this dye dispersion slowly in 5-10 minutes at 200° F.

4) Run at 190-200° F for 20-30 minutes, cool to 160-170° F, or add salt where necessary for exhaustion.

5) Flush, oxidize, soap-off and finish in conventional manner.

NOTE: In all three of the above methods it is advisable, when using any of the dyes which are known to be unstable to elevated temperatures, to disperse 3-8% WC Dextrine in cold water and add just before the point of initial dyestuff reduction.

It is to be noted that, in Method A, a gradual reduction of the dyestuff to the basic sulfoxylate leuco is accomplished slowly by a gradual rise in temperature until the dyes are completely reduced. Since the sulfoxylate leucos do not exhaust so rapidly as the normal alkaline hydrosulfite leucos, the exhaustion rate is slowed and dyeing occurs at a desired rate until the optimum temperature is reached. At this point dyeing is carried out until the desired degree of exhaustion is obtained.

This method is recommended for dyes which exhaust at a medium rate in light to medium depths and for rapidly exhausting dyes in heavy shades. If the construction of the goods causes a difficult penetration problem, Method C is preferable.

In Method B, the first stage of the dyeing is carried out with sulfoxylate as the primary reducing agent, and the majority of the amount of dyestuff is absorbed by the fiber in the first stage. The purpose of the small amount of hydro added at the final stage is to obtain the maximum color value from the dyes. This method is recommended for heavy shades where the sulfoxylate leuco of the dyes used does not give full color value.

In Method C, the low amount of reducing agent in the first stage of the dyeing results in the slow formation of the leuco at high temperature, thus promoting penetration, as the dyestuff and sulfoxylate are slowly added together.

Figure 1 illustrates the improved penetration obtained on sewed-top mercerized anklets using Method A with sulfoxylate as compared with the hydro. With sulfoxylate the dyeings were made in two cycles—first, gradually raising the temperature to 200°F for 10 minutes, and then, cooling to 170°F and running for 15 minutes. The hydro dyeings were made according to the conventional hosiery dyeing method No. 1 outlined in AATCC Monograph No. 2, "The Application of Vat Dyes". All dyeings were made on goods of the same construction using the same quantity of dye for each method.

Table I provides a partial list of those dyes which have been found to be satisfactory for any of the general sulfoxylate dyeing methods. Those marked "O" function particularly well in the overdyeing

TABLE I

DYESTUFFS USED SATISFACTORILY IN EXPERIMENTS

Colour Index or Prototype No.	*Dyestuff*	*Use**	*Reduced Color*
Pr 9	Anth Vat Yellow GC	(O)	violet
1118	Anth Vat Yellow G	(O)	royal blue
	Anth Vat Yellow PGA	(O)	red-brown
	Anth Vat Fast Yellow	—	claret
Pr 291	Anth Vat Golden Yellow GK	(O)	claret
	Veranthrene Yellow GGFW	—	blue
1132	Anth Vat Yellow GKR	(O)	red
1217	Indigoid Vat Orange R	—	yellow
1099	Anth Vat Golden Orange G	—	crimson
	Veranthrene Brill Orange GR	(O)	green
Pr 106	Indigoid Vat Scarlet 2B	—	yellow
Pr 107	Indigoid Vat Scarlet 2G	—	yellow
Pr 449	Anth Vat Scarlet R	(O)	red-violet
Pr 109	Indigoid Vat Pink FF	(O)	yellow
	Ahcovat Pink RLS	(O)	blue
Pr 296	Anth Vat Red FBB	(O)	dull brown
1162	Anth Vat Red BN	(O)	wine-red
1135	Anth Vat Brill Violet RKN	—	red brown
1104	Anth Vat Brill Violet 4RN	(O)	blue
	Indanthrene Violet FFBNWF	(O)	blue
1099	Anth Vat Dark Blue BO	(O)	violet
1100	Anth Vat Navy Blue RT	(O)	sky blue
1184	Indigoid Vat Blue 4B	(O)	yellow
969	Hydron Blue	—	dull yellow
1112	Anth Vat Blue GCD	—	greenish-blue
1113	Anth Vat Blue BF	—	greenish-blue
1114	Anth Vat Blue BCF	—	greenish-blue
	Anth Vat Navy Blue	—	blue
	Indanthrene Blue CLNWP	(O)	violet
	Anth Vat Blue-Green Y	(O)	reddish-blue
1173	Anth Vat Blue Green FFB	(O)	blue
1101	Anth Vat Jade Green	(O)	royal blue
Pr 293	Anth Vat Olive Green B	(O)	black-blue
	Ahcovat Brill Green 7G	(O)	red-brown
	Anth Vat Olive T	(O)	gray
	Mayvat Olive GGL	(B)	red-navy
	Anth Vat Gray R	—	brown
	Anth Vat Gray 2G	—	greenish-gray
	Anth Vat Gray 2RT	—	red-brown
	Veranthrene Gray BG	(O)	dull violet
	Veranthrene Gray HBR	—	red brown
Pr 121	Indigoid Vat Brown G	(O)	yellow
	Ahcovat Brown 4R	—	black-brown
	Indanthrene Brown GGA	(O)	wine
	Anth Vat Brown UF	(B)	red-brown
	Anth Vat Brown DRM	(B)	yellow-brown
	Cibanone Yellow Brown G	(O)	red-brown
Pr 125	Anth Vat Yellow Brown 3G	—	dull brown
	Mayvat Khaki BGR	(B)	red-brown
1102	Anth Vat Black BBD	—	reddish
	Indanthrene Direct Black CHF	—	red-navy

* No marking in this column indicates the dye is satisfactory for general use; "O" marking indicates that the dyestuff is particularly recommended for the overdyeing methods; and "B", for dyeing by Method B only.

Those dyestuffs with Colour Index or prototype numbers are listed with the standard name as given in AATCC Monograph No. 2 "Application of Vat Dyes", from which the various manufacturers' brands or prototypes can be obtained.

methods, and those marked "B" are recommended only for Method B. It was noted that Anth Vat Khaki 2G (Pr 122) does not reduce well by any of the general methods A, B, or C, requiring larger quantities of hydrosulfite for full depths. The recommended procedures can not be considered as optimum but should serve as a guide in setting up laboratory and production trial formulae to be adjusted to individual dyeing requirements and conditions.

From a comparison of sulfoxylate dyeings with hydro control dyeings, the following additional observations were made:

Some dyestuffs that gave unsatisfactory results with hydro under certain conditions could be dyed simply and satisfactorily with sulfoxylate under the same conditions. This was particularly true of light shades with the most highly substantive dyes of the "IN" group, such as Anthraquinone Vat Jade Green (CI 1101) and other rapidly exhausting dyes like Anthraquinone Vat Gray R.

Increased color value was obtained with several dyestuffs, for example: Indigoid Vat Scarlets 2B (Pr 106), and 2G (Pr 107), Anthraquinone Vat Navy Blue, Hydron Blue (CI 969), Anthraquinone Vat Blue BF (CI 1113), and Anthraquinone Vat Dark Blue BO (CI 1099).

Distinctly different oxidized vat shades were noted with several dyes, for example: Indigoid Vat Orange R (CI 217), Indigoid Vat Red 3B (CI 1212), Anthraquinone Vat Yellow R, Anthraquinone Vat Dark Blue BO, the indanthrones (CI 1112, 1113, 1114, and 1106), Anthraquinone Vat Olive Green B (Pr 293), Anthraquinone Vat Gray R, Anthraquinone Vat Black BBD (CI 1102), and Anthraquinone Vat Brill Violet 4RN (CI 1104).

A significant loss in color value was noted for many dyestuffs, particularly in heavy shades, as for example: Anthraquinone Vat Yellow GC (Pr 9), Anthraquinone Vat Yellow R, Anthraquinone Vat Orange RRT (CI 1098), Anthraquinone Vat Red BN (CI 1162), Indigoid Vat Red 3B, Anthraquinone Vat Olive R, (CI 1150), Anthraquinone Vat Brown G (CI 1152), and Anthraquinone Vat Khaki 2G (Pr 122).

Distinctly different leuco colors were noted with several dyestuffs, either at an intermediate stage, or at final reduction stage, for example: Anthraquinone Vat Blue BF, Anthraquinone Vat Yellow GC, Anthraquinone Vat Dark Blue BO, and Anthraquinone Vat Black BB (CI 1102).

DISCUSSION

GENERAL ADVANTAGES OF SULFOXYLATE METHODS ——— On the dyeings made with sulfoxylate, the quality was excellent, and there appeared to be no serious economical or processing disadvantages. Advantages, in addition, to slowed exhaustion rate in producing good levelness were thought to be increased stability of the reducing agent and elimination of the need for testing the reducing condition of the bath at any stage after the initial reduction. It is to be noted that, in dyeing by the pigment method, which appears to be preferable over the reduced method, it is perhaps more critical than with hydro dyeings to pigment with a good dispersion, since agglomerated dyestuff particles are more difficult to reduce with sulfoxylate.

Though reduction potential is affected by alkalinity, it was not found to be a critical factor in attaining sufficiently high reduction potentials but appears to be a general factor influencing the affinity of the leuco and, consequently, the depth of shade. The effect of temperature and time on exhaustion are also related to alkalinity. The variations in the apparent effect of these three factors for the application of individual dyestuffs preclude the stating of optimum general relationships. However, optimum alkalinities are comparable with those set forth as optimum in hydro dyeing, and can be used as a general guide. With reference to affinity, optimum temperatures, on the other hand, vary from those established for hydro in the case of individual dyestuffs. Since reduction does not occur at low temperatures with sulfoxylate, the dyeing temperature ordinarily is indicated as above the initial reduction temperature, but, in some cases, after reduction, dyeing may be carried out at a lowered temperature. An inherent characteristic of most of the leucos formed with sulfoxylate is a slower exhaustion rate, and consequently more dyeing time is required than with hydro. This is no problem in light shades, but in heavy shades dyeing time must be lengthened and usually cooling is required to obtain full depth.

Dyeing was limited to a period that was considered within practical and economic reason. In the case of some dyestuffs, which did not approach full color value within practical time limits, full depth was obtained in most cases by the addition of small quantities of hydrosulfite for a very short time at the end of the dyeing cycle (Method B). Under such conditions in general, the desired depth is obtained even if the hydro is consumed before the end of the cycle, the depth being retained and the dyestuff being kept in reduction by the action of the sulfoxylate. In dyeing compound shades in which two or more dyestuffs of widely varying substantivities were used, the variations in the exhaustion rates appeared to be minimized with sulfoxylate.

Observations made during the progress of the dyeings show that, once the near-optimum quantities of dyeing reagents and the initial reduction potential have been determined, the dyeing reduction potential and the rate of exhaustion can be varied at will by temperature and pH control. Though the initial reduction potentials may well be above the range of the particular dyestuff as established in a hydro system, and also high enough to be considered above the apparent leuco potential for the sulfoxylate system, immediate reduction does not take place as in the hydro system. Not only is the dyeing itself a function of time-temperature, but the change to the leuco form is also to a very great degree a function of time as well as of temperature. This phenomenon is indicated by the fact that, in a system in which a high reduction potential has been developed at a constant temperature, no immediate reduction is seen, but a gradual reduction takes place, as observed by the change to the leuco color. This change has been observed in some systems first on the pigmented goods, then on the dispersed particles; in other systems, the dispersed particles, not dispersed on the fiber, appear to be reduced before the deposited particles. It is possi le that this is due to variation in particle size or dispersibility.

The excellent penetraton and levelness attained is apparently due to the low initial rate of exhaustion. In a batch-type operation two factors are to be considered in explaining the levelness and penetration notwithstanding the rapid exhausting and poor leveling properties of some of the dyestuffs. The first is the slow rate of reduction of the dyestuffs at the initial leuco potential; the second is the apparently slower initial rate of exhaustion, which appears to be characteristic of the sulfoxylate systems.

pH FACTOR ——— Our experiments show that the acidity of dyebaths from the decomposition of sulfoxylate is not so high as the acidic sodium bisulfite formed by the decomposition of hydrosulfite. Sodium sulfoxylateformaldehyde solution may be considered stable to the action of acids down to pH 3. Our observations were made on blank vats with sulfoxylate versus hydro in which redox potentials were studied at varying pH values and in which the increased stability of sulfoxylate against hydro at low pH values was also demonstrated.

The increased stability of sulfoxylate compared with hydrosulfite in acid medium is graphically illustrated in Figure 7, S C Section Intersectional Contest Paper *(1)*, in which the effect, on redox potential, of lowering the pH at 200° F is seen. Verification of the high acidity of the hydrosulfite decomposition products is

shown by the fact that equivalent pH is reached with hydro at about half the addition of the acidic component as compared with sulfoxylate.

Initial reduction of the redox potential is greater with hydro at the same pH, and, while the potential of the sulfoxylate is well stabilized at a desirable value, the potential of the acidified hydro remains stable only for a few minutes and then takes a rapid drop out of the desired range, similar to the drop which is characteristic of normal alkaline hydrosulfite leuco baths when permitted to go out of reduction. Under this latter condition it is possible that the increased stability of the sulfoxylate reduction bath is due to the evolution of decomposition products of lower acidity as compared with hydrosulfite.

INCREASED DYESTUFF STABILITY AT HIGH TEMPERATURES———Laboratory experiments showed that those dyestuffs which are unstable at high dyeing temperatures, 170-212° F, in normal alkaline hydrosulfite medium exhibited a marked increase in stability when sulfoxylate was used as the reducing agent. This was found to be particularly true of the halogenated indanthrones, such as Anthraquinone Vat Blues BF, BCS, and GCD, (CI 1113, 1112, and 1114). The net result from using sulfoxylate to replace hydro was increased color value and brighter shades equal to or better than those obtained with hydro at very low, 120-30° F, dyeing temperatures. The levelness and penetration were superior because of the use of high temperatures. This phenomenon was clearly illustrated in the S C Section Intersectional Contest Paper *(1)* in which dyeings were made with four dyes of the indanthrone group on both mercerized and unmercerized cotton. The stabilizing effect was more pronounced in the dyeings in which no leuco stabilizer was used, but the differences were obvious on the dyeings in which each of the two stabilizers, sodium nitrite and WC dextrine, were used.

It is seen that, with sulfoxylate, there is no evidence in the oxidized shade of gross overreduction resulting in the irreversible formation of the tetrahydro derivative. This is further confirmed by the observation that the tetrahydro derivative was formed and appeared in noticeable quantities in many of the hydro dyeings, including those with each stabilizer, as a yellowish-brown substance with no affinity for cellulosic fiber, while in the sulfoxylate dyeings there was no visual indication of its formation. The use of sulfoxylate to replace hydro as the reducing agent in high-temperature application is therefore set forth as an improved method *(4)* for dyeing the indanthrones and other classes of vat dyes, both with and without leuco stabilizers.

OVERDYEING AND REDYEING APPLICATIONS ON COTTON

The problem of overdyeing ingrain stripes, wrap patterns, and the like, is of little concern to the dyer if he does not wish to use vat colors. However, more and more buyers are specifying 100 percent vat dyes on even pastel shades and the practice of overdyeing ingrain patterns (knit of vat- and naphthol-colored yarns) with vat colors is not unusual. Other than by using sulfoxylate, the only practical way to overdye vat-on-vats or naphthols is to use soluble vats. By using sulfoxylate, it is possible to use the cheaper conventional vat dyes without affecting the shade of the ingrain vat or naphthol pattern. As with soluble vats, the pattern will absorb the overdyed color, and it must be understood that this is not a resist dyeing method where the pattern is a medium to heavy shade. For example, a yellow may be dyed over a royal blue stripe with little or no apparent effect on the shade of the stripe and the stripe will not bleed into the body of the fabric.

The general method for overdyeing *(4)* is to wet out the goods well, then set the dyebath with a dispersing agent and 2% caustic soda on weight of the goods (0.10% on bath concentration), adding 1-2% sulfoxylate and heating bath to 200° F. The dyestuff is dispersed with an additional 1-3% sulfoxylate, previously dissolved, at 160° F, and this dispersion is added slowly to the dyebath in 5-10 minutes at 200° F. The dyestuff is reduced and exhausted rapidly and exhaustion is usually complete in 2-5 minutes after the last addition. The dyeing is then dropped, flushed, and oxidized in the conventional manner.

This method may also be used to make redye additions on vat dyeings without affecting the original shade. A variation of this method can be used for a direct redye addition to the original vat dyebath, provided a satisfactory method of quick sampling and oxidizing of the sample is available. In this method the redye addition can, in some cases, be made best by adding the dispersed dyestuff to the reduced dyebath and in other cases by a prereduction of the additive dyes with sulfoxylate. In either case it is most important that no hydrosulfite be present at the time of addition, as it would promote too rapid exhaustion. The addition must therefore be made either to a sulfoxylate dyeing or to a sulfoxylate-hydro dyeing in which the hydro has completely decomposed, as evidenced by a negative test with Vat Yellow G paper. It is not possible to obtain level dyeings under all conditions by this latter method, but too rapid exhaustion usually can be overcome if the dyebath is permitted to cool to the temperature of initial sulfoxylate reduction (140-170° F, depending upon the dyestuff) before the gradual addition is made. A second, or alternative, method *(4)* is, after reduction of the additive dyes, to adjust the dyebath with monobasic sodium phosphate or sodium bicarbonate to a range of 9.5-10.5, at which the reduced dyestuff is slowly added, and dyeing is continued to exhaustion. The success of these applications is obviously dependent upon the fact that, at low alkalinity, sulfoxylate reduces dispersed pigment rapidly, particularly in small quantities, and, under such conditions, the leuco which has been absorbed by the fiber, as well as oxidized vat dyestuff on the fiber, is not reduced sufficiently to be redistributed as the leuco.

Figure 2 illustrates examples of overdyeing by the general method detailed above. The "stripes" in the mercerized cotton tubing were attained with the following dyes: Anth Vat Navy Blue RT; Anth Vat Blue BF; Anth Vat Jade Green; Naphthol AS-SW and Scarlet R Salt; Naphthol AS-SW and Red RC Salt; and Naphthols AS-SW, AS-SG, AS-LG and Red B Salt. The dyes used for each of the overdyeings were applied at 0.5% depth owf.

APPLICATION TO FIBERS OTHER THAN COTTON

REDUCTION OF VAT TO ACID LEUCO———The increased stability of sulfoxylates as compared with hydrosulfite at all temperatures and over a wide range of alkalinity and acidity was found to be advantageous in the production of acid vats or hydrolyzed leucos and applications thereof *(4)*. Moreover, the hydrolyzed leucos formed with sulfoxylate appear to be more desirable in physical and chemical properties than those in which hydro was used as the reducing agent. Both the absorption and stability of the sulfoxylate acid leucos is better, this phenomenon perhaps being due to difference in particle size or solubility and to an equilibrium relationship between intermediate hydrolyzed stages. Another possible explanation could be a hydrotropic effect on the acid leuco by the sulfoxylate solution. The experiments indicate, by affinity variances, that the sulfoxylate vat acid is different from the hydrosulfite vat acid.

It was found that added color value was obtained, in some cases, by a partial initial dyeing of the fiber with the basic leuco, then converting it to the acid leuco

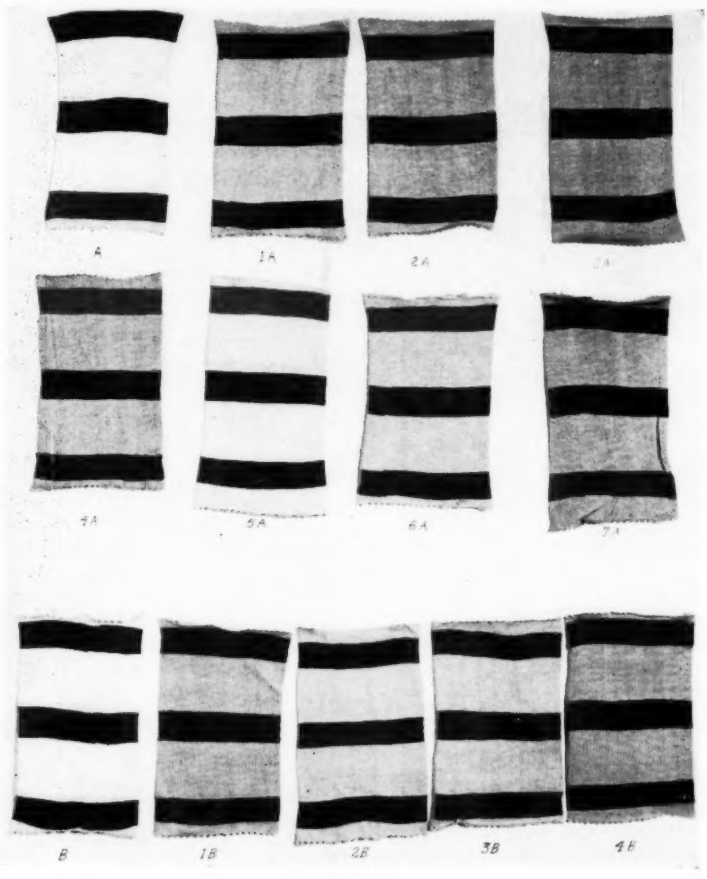


Figure 2
Examples of Overdyeing

A—Undyed goods with Naphthol stripes to show original colors.
B—Undyed goods with vat stripes to show original colors.
1A—Anth Vat Jade Green.
2A—Indigoid Brill Blue 4BR.
3A—Anth Brill Violet 4RN.
4A and 1B—Indigoid Vat Scarlet 2G.
2B and 5A—Anth Vat Yellow G.
6A and 3B—Indigoid Vat Pink FF.
7A and 4B—Indigoid Vat Brown G.

until exhaustion was reached—as opposed to the more direct process of dyeing in all stages with the acid leuco or with the basic leuco throughout the process. It was established that, for many dyeing conditions, the minimum pH must be in the range of 7 to 8, and a loss of color value is noted if the final pH is permitted to drop further. With some dyestuffs it appeared to be possible to obtain a stable acid leuco in one step directly from the oxidized form of the vat, rather than to obtain the normal sodium enolate or alkaline leuco form first and then, by acidification, to convert to the hydrolyzed leuco. In all cases, the acid leucos obtained with sulfoxylate, either by the direct method or by acidification of the normal alkaline leuco, evidenced increased stability over a wider pH range and a wider temperature range than those in which hydrosulfite was used. The application of acid leucos and partially hydrolyzed, or intermediate, leucos, without subsequent alkaline reduction, was studied, and the applications *(1, 4)* are briefly discussed in the foregoing sections. It is to be noted that, with sulfoxylate, stable acid leucos can be formed and maintained at a pH of 8.5 or slightly above this value. In the dyeing of acid vats it is appropriate to emphasize that absorptive properties of the various fibers and substantivities of the hydrolyzed leucos are largely functions of the pH factor.

During the latter stages of this investigation, it was determined that, not only for cellulosics, but for the non-cellulosics tested, the pH factor was of great importance in approaching optimum absorption of dyes, especially in the instances of individual dyestuffs which exhibit poor affinity and the various fibers which possess poor absorption characteristics. Satisfactory dyeings of good depth were obtained by variations of the alkaline sulfoxylate, vat-acid sulfoxylate, and "intermediate hydrolyzed" sulfoxylate methods *(4)* under the following conditions, which apply in general to both indigoid- and anthraquinone-type vat dyes.

VISCOSE RAYON ——— Because of the great affinity of most vat dyes for viscose rayon and of the swelling action of alkalis *(1)*, the sulfoxylate methods *(4)* without hydro, Methods A and C appear to be the most promising for knit goods by virtue of their ability to control exhaustion and of the ease with which the pH may be controlled with maintenance of the desired reduction potential. The problem of attaining color value encountered in the dyeing of cotton with certain dyestuffs was not found to be so difficult in the dyeing of viscose. However, the most easily workable pH ranges were found to be critical and varied with individual dyestuffs, but in general, the optimum pH range for indigoid types was 7.5 to 8.5 and for anthraquinones, 9.0 to 10.0 for maximum affinity. For batch-type knit-goods application, near optimum conditions appeared to be as follows:

Pigmentation at 130-140° F.
Addition of small quantities of caustic soda, buffered with sodium bicarbonate in sufficient quantity to effect reduction of pH value together with the sulfoxylate.
Moderately gradual rise in temperature to 160-190° (varying with dyestuff).
At point of complete reduction, acetic acid or monobasic sodium phosphate is added very slowly to reduce the pH to the optimum ranges noted above.

Thus, a gradual exhaustion takes place in two stages: during the initial reduction as the temperature is raised, and during the dyeing at 160-190° F as the pH is lowered.

NYLON ——— Full shades were obtained on nylon by a sulfoxylate vat-acid method *(4)*. The dyestuff is dispersed, and the goods are first pigmented at 180-200° F. Caustic soda (10%) and 15-20% Discolite (sulfoxylate) are added, and the goods are run for 5 to 10 minutes. The pH is then reduced to 6-7 by addition of acetic acid or monobasic sodium phosphate to convert the basic leuco to the acid leuco on the fiber. Dyeing is carried to exhaustion at 180-200° F. The dyeing is then rinsed and oxidized with 4% acetic acid (56%) and 4% hydrogen peroxide (30-vol) at the boil for 15 minutes. These dyeings are superior to controls run with both alkaline sulfoxylate alone and with both basic and acid hydrosulfite.

DYNEL———Good results on Dynel were achieved with the same procedure as for nylon with the addition of 2-5% of a swelling agent, such as Dowicide A (sodium o-phenylphenate) *(4)*. Data, however, were insufficient to establish this

method as a recommended or optimum one.

DACRON, ORLON 42, and ACRILAN ———Full depths were obtained on Dacron polyester fiber, Orlon 42, and Acrilan at temperatures of 204-210° F by the following method *(4):* Goods are pigmented with dispersed dyestuff at 205° F, and 5 to 10% of a carrier, and the pH is raised to 10.5-10.8 by the addition of small quantities of dissolved caustic soda and an excess of sodium bicarbonate. Reduction is carried out for 10 to 12 minutes with 15 to 20% of Discolite, and then diluted sulfuric acid is added to reduce the pH to approximately 7. Dyeing is continued at 205° F for 10 minutes, alkali is added to raise the pH to 9.5-10.0, and exhaustion is effected in 20-30 minutes. The goods are rinsed and oxidized with 2% sodium perborate and 2% acetic acid (56%). The shades obtained in this manner were considerably heavier than those obtained by various methods with hydrosulfite.

An alternate method, which does not ordinarily require a carrier, may be used where equipment is available for high-pressure dyeing. It consists simply of pigmenting first, or adding the reduced dye at 200°F, at a pH range of 9.5-10.5 (using caustic soda and an excess of sodium bicarbonate) with 15-20% sulfoxylate, raising the temperature to 230-250°F, and dyeing for 30-60 minutes.

The shades obtained on each of the acrylic fibers by either method were found to be considerably better, insofar as color value and quality, than those obtained by various methods in which hydrosulfite was used as the reducing agent.

Figure 3 illustrates the results of the dyeing method given, using temperatures below the atmospheric boil.

UNIONS———Variations of the "intermediate" hydrolyzed leuco method gave promising results on unions of cotton with the various synthetics listed above (except wool and Orlon 41) and on unions in which no cotton was present. As the critical factor for each union problem was the pH range, optimum results are obtained only with very careful control of pH values throughout the dyeing processes. Specific recommendations for various combinations are not made in this paper because of insufficient data, but it is anticipated by the author that they will be published in the near future.

THEORY

The significant differences in physical and chemical properties as well as behavior under dyeing conditions between the leuco forms produced with hydrosulfite and those produced with sulfoxylate are explained in the South-Central Section's Intersectional Contest Paper *(1)* by several possible hypotheses. The existence of a resonance relationship in the molecule of the sulfoxylate leuco in which the stress is on a different form from that of the hydrosulfite leuco is considered a possibility. A more likely explanation of the various phenomena encountered is that, with sulfoxylate, a smaller number of keto groups is reduced, and thus a different leuco molecule is produced. Molecular rearrangement appears to be a possibility with some of the dyestuffs used in the experiments. In the case of the acid leuco forms produced by the two reducing agents, there are indications that the higher reduction potentials developed with sulfoxylate at low pH values might have produced a conglomeration of three leuco or reduced forms—the enolated or basic sodium salt, the hydrolyzed or acid form, and perhaps a third or intermediate form, in which the molecules would contain some hydrolyzed keto groups and some enolated keto groups. Thus, the "intermediate" leuco might be a mixture of various leucos of a single compound of different molecular constitution. The hypothesis *(1)* further postulates the possibility that the differences in properties, particularly adsorption and affinity variations, may be due to the hydrotropic effect of the decomposition products of sulfoxylate.

Figure 3
Dyeings on 100% Dacron Anklets
(L to r): Anth Brill Violet 4RN Dbl, 3.0% owf
Anth Vat Blue-Green Y Dbl, 5.0%
Anth Vat Navy Blue RT Dbl, 5.0%
Indigoid Vat Brown G, 6.0% owf
Indigoid Vat Pink FF Dbl, 2.0% owf

SUMMARY

General methods for vat-dyeing mercerized and unmercerized cotton knit fabrics, in which sodium hydrosulfite is replaced by sodium sulfoxylateformaldehyde as the primary reducing agent, are detailed and discussed. The advantages of the sulfoxylate methods are due to the fact that the initial dyeing rate or rate of exhaustion can be controlled better and more simply than with hydro dyeings by means of temperature and/or pH control. The high-temperature stability of many vat dyes that are classified as unstable at elevated temperatures, particularly the indanthrone group, was found to be improved when sulfoxylate replaced hydro. In the experimental work no significant disadvantages were found in the sulfoxylate methods with the exception of the observation that, with certain dyes in heavy shades, the depth was not equal to the hydro control dyeings, and of the further observation that a good dispersion of the vat dyestuffs prior to reduction and dyeing is more critical than with hydro. The first disadvantage, the decrease in affinity of the sulfoxylate leuco of some of the dyes, can be overcome in most cases by the addition of a very small quantity of hydro at the end of the dyeing cycle, or by a slight reduction of the pH value of the dyebath at the end of the dyeing cycle.

In the production of acid leucos, sulfoxylate appeared to offer advantages over hydro in that pH control was easier, and the acid vat was more stable in high-temperature applications. Two practical methods of overdyeing vat and azoic effect fabrics were found in which the sulfoxylate reduction bath, under properly controlled conditions, does not affect the underlying pattern. By a variation of the same method, vat-redye additions can be made very simply without any loss in the color of the original vat dyeing.

In applying vat dyes to various synthetic fibers, including Dacron, Dynel, nylon, Acrilan, Orlon 42, cellulose acetate, and blends with cotton, dyeings made with sulfoxylate are, in practically all cases, improved over those obtained with hydrosulfite and show promising possibilities. The apparent reason for the improved results is that a greater affinity of the dyes and increased absorption by the fibers are exhibited at normal temperatures, if the dyeing is carried out in

several stages at varying pH values. The experiments have shown that the increased stability of the sulfoxylate itself as compared with hydro and of the sulfoxylate leuco form at varying pH values and at high temperatures enable the pH values of the dyebaths to be adjusted simply, and the methods to be practically adapted to dyehouse routines with a minimum amount of control, once the procedures are established.

It must be emphasized that the results contained in this paper should be considered only as a progress report on the investigation of the methods described. One important aspect of this study, which would be invaluable but which was not possible to complete in the time allotted, was the spectrophotometric analysis of the leucos obtained and the correlation of these data with the other significant factors in the reduction and dyeing cycles, such as redox potentials. Such an analysis, as described by Royer *(5)*, is anticipated in connection with future work. The investigation is being continued, and it is possible that many of the recommendations must be modified in the light of further findings.

CONCLUSION

The various methods described, wherein sodium sulfoxylateformaldehyde replaces sodium hydrosulfite as the primary reducing agent, offers the following advantages in the application of vat dyes to knitted fabrics dyed in batch-type processes:

a) Controlled rate of exhaustion, resulting in more level and better-penetrated dyeings.
b) Increased stability of vat dyestuffs at elevated temperatures, resulting in brighter, truer shades and in better matches from lot to lot.
c) Increased stability of the reducing agent, resulting in simpler control of dyeing procedures.
d) A practical method of overdyeing vat and naphthol stripes and patterns and of redyeing off-shade dye lots without affecting the base shades in either case.
e) Promising possibilities in the dyeing of fibers other than cotton, including blends of mixed fibers, at dyeing temperatures below 212° F by means of the control of the pH factor during reduction and dyeing.

The principal disadvantage encountered was the problem of dispersibility of the dyestuffs as supplied by the various manufacturers. It is understood that each manufacturer might use different dispersing agents in some of his dyes and undoubtedly he uses different dispersing agents than some of his competitors on prototype dyes. The ideal reduction condition for sulfoxylate is dependent upon good dyestuff dispersion, and the premature agglomeration of dyestuffs prior to reduction can result in poor dyeing quality, though this is considerably more important in package dyeing of yarn then it is on hosiery or knit goods dyeing. At the time of the completion of this paper, an ideal dispersing agent for all dyes had not been found and the possibility is recognized that, due to interfering agents in various dyes as marketed, it might be extremely difficult to find any one dispersing agent which will be universally good.

The problem of surface oxidation of dyestuff and deposition of oxidized dyestuff on dyeing vessels has also been recognized, though this has not caused any serious difficulties.

QUESTIONS

Question: Can you bring vat dyes into clear complete solution as vat leucos with formaldehydesulfoxylate?

Answer: Yes.

Q: Can you make a filter test with such a leuco solution?

A: Yes.

Q: Do you have other evidence, besides behavior toward fibers, that sulfoxylate leucos differ from hydrosulfite leucos?

A: Yes, not only do they differ in affinity at the same pH and temperature, but they differ in color.

REFERENCES

(1) South Central Section, AATCC, *Am Dyestuff Reptr* 43, P525 (1954).
(2) AATCC Monograph No. 2, "The Application of Vat Dyes", (1953).
(3) Clark, O W, *Am Dyestuff Reptr* 38, P 828 (1949).
(4) Blum, Robert D, Jr, Patents applied for.
(5) Royer, G L, *Am Dyestuff Reptr* 41, P 597 (Part II) and *Calco Technical Bull No.* 828.

ACKNOWLEDGMENTS

The co-operation of the following in furnishing essential equipment and other valuable assistance necessary for the completion of the experimental work in this paper is gratefully acknowledged:

Royce Chemical Company
The Foxboro Company
Macbeth Corporation
P J Wood

NOTE: A large portion of the experimental work of this paper was carried out simultaneously with experimental work performed for inclusion in the 1953 Intersectional Contest paper of the South-Central Section, AATCC, "Investigation of Sodium Sulfoxylate-formaldehyde as the Primary Reducing Agent in Vat Dyeing", for which the author served as Intersectional Contest Committee Chairman. It is to be noted that some of the data and discussion from the brader subject matter of the contest paper, which were deemed pertinent to the specialized subject matter contained in this paper, were included in order to make the presentation herein as complete as possible without an excess of cross references.

AATCC Monograph No. 2
"THE APPLICATION OF VAT DYES"

"The group of fifteen editors responsible for this monograph consists of a well-balanced cross-section of American industry concerned with the production, marketing and application of vat dyes. The AATCC is to be congratulated on the production of a comprehensive survey of American vat dyeing and printing practice which is the major theme of this application . . . the book will be useful as a work of reference at this side of the Atlantic and as a working manual for the American colorists for whom it was designed. . . . If a clear account of American practice is required . . . the enquirer *must* have access to this volume."—*JOURNAL OF THE TEXTILE INSTITUTE* (England).

"This book . . . is abreast of the latest technical developments and research. Its value is increased because of the lack of books in this field today . . . we can readily recommend this book to all technical and practical men"—*MELLIAND TEXTILBERICHTE* (Germany).

"This monograph contains the work of many mill-experienced authors, plus good, sound chemical and application principles developed in the laboratories and mills enjoying AATCC membership. . . . This book puts between two covers much information that a dyer is constantly searching for. It is a good investment for any laboratory, dyehouse, or printshop."—*TEXTILE WORLD.*

". . . this monograph should be in the hands of every user of vat dyestuffs, every student and every teacher. The book is well indexed and includes valuable lists of equivalent trade names. It will certainly take its place as a standard book of reference."—*JOURNAL OF THE SOCIETY OF DYERS AND COLOURISTS* (England).

$5.00, postpaid, to members
$6.00, postpaid, to nonmembers

SEND CHECK OR MONEY ORDER TO

DR H C CHAPIN, SECRETARY, AATCC
PO BOX 28 ● LOWELL, MASS

New York Section——

A REVIEW OF TEXTILE-SHRINKAGE CONTROL PROCESSES AND TEST METHODS*

JOHN F WARNER
D B Fuller & Co, Inc

The phenomenon of fabric shrinkage is explained by the author in general and is applied particularly to cotton woven fabrics. The compressed shrinkage of cotton materials to control shrinkage in subsequent use is discussed, and the use of resin finishes to control shrinkage is mentioned.

The development of the present method of testing the shrinkage of cotton fabrics and the work being carried out internationally and more especially by the AATCC to improve that method and to bring international agreement are covered.

INTRODUCTION

FOR the next few minutes, I suggest we stand to one side, as it were, and take a good look at fabric shrinkage, which we know is one of the most important problems of the textile manufacturer. The subject of shrinkage has been thoroughly covered; for example, as far back as 1948, a bibliography prepared by the US Quartermaster Research & Development Laboratories listed a total of 242 articles by 171 authors *(1)*. Since that time the list has been greatly expanded. I am sure you will agree that the subject of shrinkage is of interest to all of us. Along with color fastness, shrinkage problems, directly and indirectly, occupy much of the time of those of us who are actively engaged in the production and sales of textile fabrics.

GENERAL

VARIOUS CAUSES OF SHRINKAGE ——Our present knowledge indicates that the shrinkage problem of textiles varies according to the nature of the fiber in the textile material, so that antishrink treatments which are quite suitable for, say, wool garments are quite unsatisfactory when applied to cotton or a cellulose rayon *(2)*. The new true synthetics also show their own peculiar behaviors in washing.

Ultimately, fabric shrinkage in washing can be traced back to the behavior of the individual fibers in wetting and drying and to the mechanical movements that are involved. As we shall see, fiber structures comes into this, and the more we know about the way in which fibers are built up from their units of structure, the better able we are to devise treatments for modifying those features that contribute to shrinkage. Thus, while a good knowledge of fiber chemistry is of great help, it has been found that methods which involve the use of x-rays and the electron microscope have also become important.

INFLUENCE OF THE FIBER——It has been mentioned that the type of fiber plays an important part in deciding the shrinkage suffered by a textile material in washing. But it is also true that all fibers possess certain common properties that induce shrinkage. So, it will be convenient to mention these before dealing with the special methods that have been evolved to make the various kinds of fabrics and garments resistant.

The two most common properties of this kind are as follows: 1) the readiness of a textile fiber to swell and thus become thicker when wet and the corresponding tendency of the swollen wet fiber to return to its previous state when dried, and 2) the strong tendency for a stretched fiber to contract to its normal length when wetted. These two properties play an important part in promoting shrinkage when textile materials are washed. We will now examine them somewhat closely.

FIBER SWELLING —— Cotton, rayon, linen, wool, and silk fibers all have a strong tendency to imbibe water when wetted, and the water thus absorbed into their interior distends their fine structure so that they become 30 to 40 percent thicker but only about 5 percent longer or shorter *(2)*. On drying, the wet fibers gradually go back to their original thickness. These changes can be repeated many times without appreciably modifying the capacity of the fibers to change in the same manner.

Let us for a moment consider a length of yarn to be made of comparatively short fibers more or less parallel to each other. If the yarn is wetted, then each fiber becomes much thicker, and the yarn thickness increases to a corresponding degree. There is no material change in length of the yarn. Now, let us consider a similar yarn but with the fibers twisted about each other, as is customary in a weaving or knitting yarn, the twist being for the purpose of holding the fibers closer together so that greater adhesion and tensile strength are obtained. When this twisted yarn is wetted, the fibers become thicker in the usual way, but, under the conditions imposed upon them by the twist, the yarn has to contract in length in order that they may become thicker. Fiber swelling is thus the cause of yarn length contraction. If this yarn forms a part of a woven or knitted fabric, then the fabric has to shrink in area in order to accommodate the shrinkage of its constituent yarns.

VARIATIONS AMONG FIBERS—— The different textile fibers vary among themselves in respect to the amount of water that they can imbibe on wetting and thus the degree of swelling that they undergo. Ultimately, it is the way in which the long-chain molecules are originally packed within a fiber which determines its behavior on wetting. In some fibers the chain molecules are very closely packed and are mainly parallel to each other; such fibers resist excessive swelling on wetting. Other fibers have their chain molecules much less parallel to each other and somewhat loosely packed, these readily swell to a considerable degree.

LIQUOR PENETRATION —— A point about the behavior of fibers, such as cotton and wool in contact with an organic solvent, is that in most cases the solvent molecules are too large to pass through the fiber cuticle *(2)*. But in the presence of water, this rule does not hold good, for the water is able to swell the fibers sufficiently to make it easier for the large molecules of the organic solvent to enter. Thus, moisture is the most important liquid which is able to bring about fiber swelling, such as can induce shrinkage in washing and laundering.

All textile fibers can be stretched, and, indeed, a fiber that would resist stretching completely would be almost useless in textile manufacture. In converting fibers into yarns and fabrics by the many processes that are required, the fibers are almost continuously subjected to some form

* Presented at Kohler's Swiss Chalet in Rochelle Park, N J, on Friday evening, April 9, 1954. The ms was recd by the Editor of Proceedings on April 1, 1954, and by the Am Dyestuff Reptr on Aug 17, 1954.

of tension, which varies considerably according to the process. By stretching to accommodate these tensions, the fiber avoids breakage. But it matters a great deal how much the fibers can be stretched and the ease with which they do so.

YARN CHANGES IN WASHING———In the washing of textile materials, yarns are pulled this way and that throughout the operation; and thus there is a tendency for them to be stretched*(2)*. This tendency is the greater because most fibers stretch more easily when wet. But in washing, a fabric or a garment which is pulled in one direction often contracts correspondingly in another; this occurs more with knitted than with woven materials. So that the effect of tension in washing is seldom sufficient to prevent shrinkage from other causes.

In washing, it is really the previous stretching of the goods being washed which is most important. Cotton, wool and similar fibers have the peculiar property that, if dried in a stretched condition, they have the power to remain set in this stretched state until they are once more wetted, when they at once return to their unstretched condition. Most of the treatments to which a fabric or garment is subjected in order to give it a desired finish or otherwise to make it attractive to the user have the effect of stretching the fibers so that the textile material is brought to an area greater than normal. Somewhere in these treatments this stretched condition is set by drying—often under pressure. Thus, finished textile materials are usually in an unstable state, so that they will shrink in washing simply by release of this strained condition.

COTTON SHRINKAGE——— WOVEN FABRICS

At this point, a short history of the development of cotton-piece-goods manufacturing and the requirements of the finisher might be interesting. When plain looms were used for weaving, the contraction of the warp amounted to from 5 to 10 percent. Such cloths, if put through the regular finishing process without special stretching devices, would gain from 2 to 3 percent. (This two- to three-percent gain means a two- to three-percent contraction during washing). The converters who received the benefits of this gain became accustomed to such an average and usually considered the gain in their price or profit calculation. Subsequent improvements in looms reduced the contraction of the warp, and, in most cases where goods from such looms are put through the finishing process without special stretching devices, the result is a loss in length, which is further aggravated by calendering and other means of stretching by the mill in the greige (unbleached) state and before finishing. Subsequent demands by converters for gains to which they had been accustomed forced the finishers to adjust their processes so as to produce gain at the expense of quality and appearance.

To sum up, shrinkage results vary from the fact that textiles are stretched in varying degrees during the entire manufacturing operation. Furthermore, since they are usually dried in a stretched condition, often in the presence of sizing, the yarns almost always maintain their length until they come in contact with moisture, as during the first washing. If the fabrics have been stretched only slightly during manufacture, the contraction during washing will be equally slight. On the other hand, if textiles have been stretched excessively during manufacture, contraction during the first washing will be serious. It was the recognition of this fact by certain progressive manufacturers that brought about the development of satisfactory preshrinking processes, such as compressive shrinking, since it was soon realized that the logical point at which to prevent shrinkage was prior to laundering. As has been aptly said, "Control of shrinkage was not the product of the inventive mind of the textile industry, it was born of necessity—an insistent demand from the consuming public—and those who contend the public will not pay for serviceable qualities in washable textiles are applying the traditional thinking of their grandfathers in the industry. . . ."

Men's shirts and work clothes, women's and children's dresses, and other items may be worn for some time after the colors have faded or the effect of the finish has disappeared, but once they become too small, they are no longer of value to the wearer.

This was graphically demonstrated a couple of years ago by a headline in metropolitan papers carrying a Milwaukee dateline, which said, "Shoots 3 Dead Because Store Shrank His Pants". Fortunately, dissatisfied customers seldom go to such extremes, but I submit the episode does carry a warning to all of us.

While a partial solution of the shrinkage problem was originally obtained through washing the goods or by moisture treatment under relaxed conditions, this had several drawbacks. The usual type of treatment which was practical in the mill at reasonable cost did not thoroughly shrink the goods, and further shrinkage was obtained when the fabric was washed by the consumer. Further, the hand and appearance the finisher had worked so hard to obtain was destroyed, leaving usually a wrinkled and raggy piece of goods, which had no customer appeal.

In spite of everything said so far, the preshrinking of cotton cloth has been worked out to a practical solution only in the past couple of decades, despite the fact that dimensionally stable cloth from the point of view of the consumer is probably more important than stability in any other quality.

Today, there are two principal methods open to the finisher for obtaining a shrink-resistant fabric:

1) application of synthetic resins to "set" the yarns and fibers.
2) mechanically forced shrinkage to the point where further dimensional change does not occur on washing.

The latter method, called *compressive shrinking* is the most widely used for cotton and will be considered first.

COMPRESSIVE SHRINKING———In virtually any country in the world that boasts a textile industry, you will find compressive shrinking machines. How did it come about that this problem of cotton shrinkage—which had been a problem literally for centuries—was suddenly solved? Perhaps, more importantly, how did the solution to the problem find such worldwide acceptance in so short a period of time?

I think, in the interest of historical accuracy, credit must go very largely to one man, Sanford Cluett, and to one firm, Cluett, Peabody & Co, Inc. Describing his invention, the inventor said: "I looked at a piece of cloth and I thought about its condition. It had been pulled lengthwise through a weaving mill and through the finishing mill, and it was stretched out of shape along its length".

"My problem was to counteract the pull. The opposite of pull is push. So I literally pushed it with my finger. But of course it simply wrinkled. Then I put it on *my* knee and pressed a stretched rubber band on the cloth, gradually letting the rubber band return to normal. The cloth, unable to wrinkle, did what it wanted to do. It shrank with the rubber band".

Sanford Cluett thus reduces to the very simplest terms the basic principle of an invention that has developed into a shrinking method that has received general acceptance by the world's leading textile manufacturers. And wherever you find compressive shrinking machines, you will find our CCC-T-191a test in continuous daily use . . . a US-developed but universally accepted procedure for determining the shrinkage performance of any specific fabric.

In commercial practice, mechanical "compressive shrinking" is carried out by forcing a dampened cloth to the dimensions desired (previously determined by shrinkage tests) and drying it while in those dimensions. The dampening with

water or steam has two purposes: to force some shrinkage by fiber swelling, and to "plasticize" the cotton sufficiently so that the cloth may be compressed lengthwise without wrinkling. The compression is carried out by bringing the cloth into contact with an expanded surface and holding it there while the surface is compressed or allowed to relax. In one process this surface is a thick blanket; in another process the surface is a stretched rubber belt.

While compressive shrinkage is still the most widely used method of controlling the stability of cotton piece goods, the use of thermosetting resins is increasing. The development of resin finishes to stabilize cottons and, incidentally, when properly applied, to also add crease resistance, reminds me of the story of the small boy who came to school one day very tired.

"Why are you so tired, Johnny?", asked the teacher.

"My stepfather has been teaching me how to swim. He took me out fifty yards and threw me into the water".

The teacher thought that was strenuous but maybe all right.

Next day, Johnny came in the same way and said he had been taken out a hundred yards. The third day, he appeared terribly worn out. This time, he said his stepfather had taken him out a whole mile.

"It must have been tough swimming all that distance", said the teacher. "Oh no", answered the boy, "the swimming was no trouble *once I got out of the sack.*"

Only in recent years have these resin finishes emerged from their sack. As is the case with all chemical developments, much progress has been made, but the field is still wide-open for improved products. Back in the early 1930's, when one or two American finishing plants started to treat cottons and viscose rayons under license from Tootal-Broadhurst-Lee Co, who owned the dominant patent, the only resins available were rather unstable urea-formaldehyde paste types. These were followed ten years later by the development of the melamine-formaldehyde resins, which gave better stability and were more wash-fast than the ureas. The use of urea and melamine resins on cottons was somewhat retarded by the fact that garments so treated picked up chlorine from wash waters, and under certain conditions turned yellow and lost strength.

Recently, however, modified urea-formaldehyde products have found general use. These materials, although not entirely nonchlorine-retentive, are nevertheless sufficiently safe to warrant general use on light and even white-ground cotton fabrics. As you all know, the application of resins to cotton piece goods is not a particularly difficult operation, and many cotton finishing plants are now properly equipped. However, because we are working with chemicals where control of product and process is important, the end result, so far as shrinkage control is concerned, is subject to wide variation. Some resin-finished cottons remain stable for only a few washings, whereas other fabrics which have been properly controlled exhibit a degree of stabilization approaching that imparted by a purely chemical modification of cellulose with aldehydes.

THE NECESSITY FOR USING CARE IN TAILORING ——— In addition to choosing fabrics that have been correctly preshrunk, the garment manufacturer finds it necessary to pay considerable attention to the tailoring methods that are used. It should not be expected that a preshrunk fabric is magically converted into a rigid material that cannot be stretched with ordinary handling. Recognizing that improper tailoring methods may stretch preshrunk goods, Cluett, Peabody & Co, Inc, for example, give the following recommendations to garment manufacturers who use "Sanforized" materials: "Fabrics that are completely shrunk are more elastic than partially shrunk or unshrunk fabrics, and therefore have a tendency to elongate when subjected to tension".

"To avoid distortion of a *Sanforized* fabric in manufacturing operations, the following suggestions should be carefully observed:

1) Make sure that cutting patterns are the proper dimensions to produce a garment of the exact finished size desired.

2) Do not pull the material while spreading it on the cutting board.

3) Do not dampen or sponge material before cutting or after placing it on the cutting board.

4) In all stitching operations, the presser foot on the sewing machine should have as little pressure as possible on the fabric.

5) The thread tensions, upper and lower, should both be run as loose as possible. Thread always shrinks upon wetting, and if the tension in the thread is tight, it tends to pucker the fabric, even though it is completely shrunk.

6) When stitching, the operator should not pull the fabric in an endeavor to make it guide evenly.

7) When the finished garment is to be pressed, a light spray only should be used. If the garment is to be ironed by hand, the ironer should not stretch the garment during the ironing process. If a machine press is used, the wrinkles in the garment should be smoothed out but the garment should not be stretched.

8) All new fabrics should be tested for potential wash shrinkage by washing before manufacturing material into garments. At least one garment should be washed and pressed in a commercial laundry to determine positively that proper size has been provided and as a final check on the material used".

LABORATORY TEST METHODS

Having briefly covered the theoretical and practical aspects of stabilizing cotton piece goods, let us now consider the various laboratory test procedures we have been using to determine whether the fabrics so treated (whether by compressive shrinkage or synthetic resins, or by a combination of both) would give consumer satisfaction.

Back in 1938, when the Federal Trade Commission established *Trade-Practice Rules for the Shrinkage of Woven Cotton Yard Goods*, it was specified that the fabric should be tested by the procedure outlined in "Commercial Standard CS59-36". This is the well-known CCC-T-191a test, which is the same as AATCC Test 14-33 and ASTM Test D437-36. In other words, this test was accepted and used by all segments of American industry for determining the shrinkage of cotton woven piece goods. In recent years it has also been adopted by Sanforized licensees in 38 foreign countries.

The reason this particular test was originally accepted was that it had been found that the shrinkage obtained by its use on woven fabric corresponded closely to the shrinkage of the same fabric when washed by the consumer, whose interests were the primary concern of the Federal Trade Commission in promulgating the original rules.

Of course, like all tests, CCC-T-191a isn't 100% perfect and has been occasionally criticized. Such criticisms of details in the test procedure, while not serious, were nevertheless recognized by the AATCC Committee on Dimensional Stability, and. inasmuch as Fed Spec CCC-T-191b, issued May 15, 1951, also differed in several respects from 191a, the Committee decided in the summer of 1952 to undertake a series of interlaboratory test methods to check the disputed points.

Incidentally, the principal changes in CCC-T-191b are 1) washing at the boil for the entire 40-minute cycle, and 2) increasing the load from 3 pounds to 6-8 pounds. Such changes would, of course, make it more difficult and costly for mills and finishing plants to test shrinkage in plant control work. Nevertheless, the Committee agreed we should try to find the facts, and if any of the changes or recommendations were found significant, we should be guided accordingly. Five laboratories co-operated, and each of the proposed changes in the tests was repeated ten times on every fabric.

While the interlaboratory work was being organized, a meeting was held in June, 1952, under the auspices of the American Standards Association for the purpose of considering a proposed international test to be used by all members of

the International Standards Organization for testing the shrinkage of cotton textiles. At that meeting the USA delegation endeavored to have the proposed international standard follow closely CCC-T-191a, but did not succeed. Several delegates questioned the load factor and time cycle and offered substitutes. Inasmuch as we serve as secretariat for this particular test project, we decided to expand our AATCC interlaboratory work to include a study of the questions raised at that Meeting. Hence, the interlaboratory test program as set up comprised testing the following fabrics: seven different constructions of cotton piece goods which were unshrunk, and the same seven cotton fabrics which had been compressively shrunk to under 1% residual shrinkage. The fabrics were representative of carded and combed shirtings, dress goods, and work-clothing fabrics, and ranged in weight from four yards to two yards per pound. We also included in the program two chemically treated viscose cloths: a light-weight challis and a medium-weight gabardine. These two constructions are representative of the principal men's shirting fabrics in their field.

The shrinkage tests comprised eight different procedures. For example, the load factor was varied from three pounds to fifteen pounds of dry fabric. Other variables included maintaining the temperature at the boil for 40 minutes in one test, for 60 minutes in another test, running the test for a total of 80 minutes instead of 60 minutes, changing the wash liquor at the end of 20 minutes, and repeating the wash cycle five times, etc, etc.

Having gone this far, the Committee then decided to have the same sets of 14 cotton fabrics and two viscose fabrics washed repeatedly (ten times) in an automatic home washer and finally to have another complete set of the same fabrics given a series of commercial launderings. The commercial laundering test was further broken down to include two typical wash-wheel procedures: 1) an average work-clothing formula and 2) a typical white-work-load procedure. We felt these additional data should provide a picture of the way in which shrinkage occurs in a fabric and especially the relation of shrinkage in domestic home laundering to shrinkage in commercial laundering, as well as how such shrinkage figures correlate with laboratory results.

We wound up, as you can appreciate, with thousands of test figures, and the Committee asked to have the results of all the tests analyzed statistically at the AATCC Research Laboratories in Lowell.

In view of the nature of the data available, ie, the seven radically different types of cotton fabrics being investigated, the difference in methods, and the different machine loads, it was decided to apply the "F" Test to the results. The "F" value is most useful in the analysis of variance and is calculated when several tests are made to determine if there is a greater difference between methods than within the method itself. It is likewise possible to determine if there exists a greater difference between laboratories or between individual specimens within a laboratory.

If the calculated "F" value is found to be less than a predetermined value at 95% probability, there is no significant difference and 95 of each 100 determinations will not vary significantly. If the calculated "F" value is greater than the predetermined value at 95% probability, a significant difference does exist. The 95% probability is generally considered a sufficiently high level of agreement for most textile testing.

Determination of the "F" value for agreement between the five participating laboratories showed that there was a significant variation among the laboratories in the results obtained in six of the seven fabrics tested by the six modifications of CCC-T-191a and CCC-T-191b. Closer examination of the individual test readings disclosed that most variation appears to be due to an undetermined cause existing in Laboratory I. Elimination of Laboratory I from the determination of the "F" value for agreement between laboratories confirmed the fact that Laboratory I is not in good agreement with the other four laboratories, since satisfactory "F" values were now obtained on four of seven fabrics and the values were considerably lower.

CONCLUSIONS

The AATCC report from Lowell concludes that, on the basis of the data available from the interlaboratory testing program, no appreciable differences in shrinkage results would be obtained to justify a change to a modified CCC-T-191a method.

Although CCC-T-191a does show effect of the machine load on certain fabrics (two out of seven), it is a much better test method than CCC-T-191b, which shows an effect of machine load in five out of seven instances.

The other procedures, CCC-T-191b with 3-pound dry load, the British and the French proposals with 15-pound loads, although showing no significant difference between the methods, give a total shrinkage which is more comparable to the lower total shrinkage obtained in domestic laundering in most instances.

The Committee believes that the standard CCC-T-191a method using a normal 3-pound dry-fabric load remains the better method, since this method gives final shrinkage results closest to that obtained in the commercial laundering. This method gave total shrinkage comparable to commercial laundering in all fabrics except the poplin fabric, even though the method is slightly less reproducible between laboratories than when a 15-pound dry load was used.

Further investigation is indicated to determine statistically the variation in results which is attributable to inherent fabric variation. A large number of individual test readings for specimens of each fabric tested by one method would permit analysis to determine if the variation was significantly greater in each fabric than the variation that might be caused by the method of test employed.

The complete report will be published in an early issue of the American Dyestuff Reporter.* To give you an indication of what the results show, however, we will quote only the warp shrinkage figures in percent shown on the carded denim at the end of ten repeated washings:

191a (3 lb)	0.8
191a (8 lb)	0.3
191a (15 lb)	0.5
191b (3 lb)	0.5
191b (8 lb)	0.3
191b (15 lb)	0.7
British (15 lb)	1.4
French (15 lb)	0.5
Commercial	—1.1
Domestic	—1.6

One final word. What we, who are active in industry, want are good reproducible laboratory tests, even though such tests may not be 100% letter perfect. For example, in the case of cottons we recognize that it no doubt would be possible to develop a laboratory test method which would show slightly more shrinkage on an occasional construction than our present AATCC test method. However, such a laboratory test would not necessarily be better from a practical viewpoint. After all, a cotton garment is almost always worn at least once after each laundering and because of the action of the body when wearing the garment, the fabric usually has an opportunity to relax slightly.

Finally, in closing, let me say the Committee on Dimensional Stability is not closing up shop. One of our next projects should be equally interesting: an exhaustive study of reasons for the shrinkage that sometimes occurs in garments manufactured from cloth which has satisfactorily passed the laboratory test, but which shrinks unduly when laundered and tumble-dried.

---

* EDITOR'S NOTE: Following the delivery of this paper by the author on April 1, 1954, the report referred to was published in its complete form. (See page P364 of the June 7, 1954 issue.)

REFERENCES

(1) Bibliography on Shrinkage, U S Dept of Agriculture, Bibliographic Series #3, Jan. 1948. Patent and literature survey on shrinkage and shrinkage control of textile fabrics. Tech Library, QM Research and Development Labs, Philadelphia, Pa.

(2) Technologist, *Textile Mercury and Argus*, Sept 23, 1949.

Rhode Island Section——

VAT DYES—A MISCELLANY*

MAURICE R FOX

Imperial Chemical Industries Ltd, England, and Arnold, Hoffman & Co, Inc, Providence, R I

The chemistry of vat dyes is touched upon, and the chemical structures of some of these products are discussed in the light of reducing, dyeing and fastness behavior. Present-day European application methods are briefly outlined.

INTRODUCTION

THE "simple principles" of vat dye application have been written up in many papers and technical publications, and it need hardly be repeated here that vat dyes carry all-important keto groupings. In order to create real affinity for the fiber in a process of dyeing or printing, such groups in the dye structure must be reduced to the enol form, usually in alkaline solution, if a happy marriage of dye and fiber is to result.

Many unhappy marriages occur, and a large number of problems which confront the dyer daily have no ready-made explanations, in spite of what the dye salesmen may say. Many of these problems can, of course, be solved at the present time, but we are a long way from a complete understanding of the mechanisms of reduction and oxidation of the vat dyes, and perhaps even further from knowing all the answers on their mode of entry and retention in textile fibers. Vat dyes are being applied as fully oxidized pigments for the mass pigmentation of regenerated cellulose and modified cellulosic fibers, but it seems that we are a long way from applying vat dyes to manufactured yarns and piece goods by any means which avoids the necessity of reducing and reoxidizing the dye. We still have to contend, therefore, with reducing agents and in particular with sodium hydrosulfite, and although this wonderful product has been employed for vat-dye application for half a century, we do not yet know the intimate details of its behavior in such a process. We can work out a variety of chemical equations and show a variety of by-products from this combination of sodium, sulfur, and oxygen, but all we really know is that it supplies hydrogen in a form which will convert quinones to hydroquinones or oxidized vat dyes to reduced vat dyes.

REDUCTION OF VAT DYES

The rate at which a vat dye is transformed into its leuco state is of the utmost importance in this age of high-speed dyeing and printing techniques. The value of a dye for such processes is largely dependent upon the rate at which it reduces. Twenty years ago the vatting operation was one of 30 minutes by necessity; today we are expected to obtain full tinctorial value and fastness behavior by processes involving reduction times of 10 seconds and shorter. An improvement in vatting speed of some 200 times faster than 20 years ago has been demanded, and in a large number of cases this has been met by the dye manufacturers. This is, of course, not wholly true, since our studies have shown that there is an important relationship between the temperature at which the reduction process is operated and the rate at which the dye reduces, and our temperatures of dyeing relative to those used 20 years ago have at least doubled themselves.

A study of the reduction of vat dyes is complicated by the fact that they are water-insoluble in the oxidized state and a heterogeneous state exists during the vatting operation. The finely dispersed pigment dye passes into solution as the reduction process is allowed to proceed. The rate of reduction must therefore be dependent to a large extent upon the physical form of the pigment itself. By physical form we mean particle size, particle shape, and crystal habit and uniformity of these attributes throughout a given amount of dye. When certain vat dyes are crystallized from solvents or dried directly from an aqueous paste without careful processing, they will not reduce by normal techniques, and it has therefore always been a target for the dyemaker to supply his vat dyes in an easily reducible form. We are learning more each day about ways and means of achieving this end, and my colleagues at Imperial Chemical Industries Ltd, Dyestuffs Division (in particular, Marshall and Peters *(1)*), have already made a detailed study of this problem from a fundamental point of view, and much of practical value has resulted from their work. The work, which continues, is revealing the extent to which the rate of reduction varies between dyes of different chemical structure and physical state, and the goal is set to determine the optimum state for the dyer and printer.

Apparatus has been designed for the purpose of studying the reduction process, and this relies on the fact that absorption spectra of the suspended vat dyes and the leuco compound usually show different extinction coefficients and different wave lengths of maximum absorption. Under constant conditions of test it has been shown that the time required for the reduction to proceed halfway to completion at 105°F ranges from less than 5 seconds up to 3000 seconds over a short range of commercial dyes. Increasing temperatures give higher rates of reduction; thus, even at 140°F the increase is approximately threefold. It is also of interest to find that the so-called cold-dyeing dyes have generally a higher rate of reduction than the hot-vatting thioindigoid types (see Table I).

TABLE I

REDUCTION RATE PROPERTIES OF VAT DYES *(1)*

Dyestuff	*CI or Pr Number*	*Time of Half-reduction at 105°F 20 g/l NaOH and $Na_2S_2O_4$ seconds*	*Recommended Vatting Temperature °F*
Caledon Red BN	CI 1162	<5	85
Caledon Brill Violet R	CI 1135	32	105
Caledon Jade Green XN	CI 1101	50	112
Durindone Brown G	Pr 121	780	175
Durindone Pink FF	Pr 109	2880	175

* Presented at the Sheraton-Biltmore Hotel in Providence on Thursday, March 25, 1954. The ms was rec'd by the Editor of Proceedings on June 10 and by the Am Dyestuff Reptr on Aug 5, 1954.

VARIETY OF VAT-DYE STRUCTURES

It would be impossible in a paper of such short duration to give a detailed picture of the behavior of all the various chemical identities used as vat dyestuffs, but it is thought that reference to a few specific examples will serve to illustrate the diverse behaviors of the vat coloring matters.

From the simpler acylaminoanthraquinone vat dyes we can choose three closely related dyes for discussion—

No. 1 *Anthraquinone Vat Yellow 5GK (2)*

No. 2 *Caledon Yellow 4G (2), Abcovat Yellow 4G*

No. 3 *Indanthrene Orange GG (2)*

These dyes are closely similar in constitution, and it should be noted that the central nucleus of all three products may be interchanged from terephthalic acid to isophthalic acid with little significant influence on the shade and properties of the ultimate product. The first product (No. 1) is devoid of benzoylamino groups and has least affinity for cellulose fibers from the dyebath; it is greenest in shade but has the lowest fastness to light of the series. This product is rarely used for dyeing purposes but finds its greatest outlet in the printing trade. In order to obtain full color value from the product, it must be dyed at low temperature with high electrolyte content and low concentrations of caustic soda.

With the addition of benzoylamino groups in the 5:5'—or the 5:4'—positions, there is a marked increase in the affinity of the dye for the fiber, and both benzoylated products are applicable by Method 2 dyeing methods, ie, 120°F. Both products, however, are redder in shade than the unbenzoylated parent, No. 3 being appreciably redder than No. 2.

Product No. 1 exerts a catalytic fading effect on the Vat Jade Greens (CI 1101), but this is much milder, if at all evident, in the two redder-shade products, and valuable yellow-green shades can be obtained in this way with No. 2 as the yellow component. All three products are sensitive to prolonged contact with high temperatures in the reduced state; fission occurs at the —NH.CO— linkage.

A parallel effect of the benzoylamino group is noted in three further products drawn from the anthraquinone carbazole group of dyestuffs:

No. 4 *Anthraquinone Vat Yellow FFRK* (Pr 450)

No. 5 *Anthraquinone Vat Golden Orange 3G* (Pr 290)

As 4, but carrying benzoylamino groupings in the 5:5'-positions.

No. 6 *Anthraquinone Vat Brown R* (CI 1151)

As 4, but carrying benzoylamino groupings in the 5:4'-positions.

Again the unbenzoylated product is greenest in shade and has the lowest affinity for cellulose. Fiber-tendering propensities decrease in the order given. The Yellow FFRK demands low-temperature dyeing conditions and a low concentration of caustic soda in order to obtain useful tinctorial yield and to avoid leuco-dye decomposition.

Flavanthrone or Anthraquinone Vat Yellow G (CI 1118) is a valuable though peculiar product which can give rise to difficulties under some conditions of application. Structurally, it is considered to be No. 7. (See formula on page P732).

This product is exceptional insofar as it has a high rate of reduction (time of half-reduction is less than 5 sec at 105°F) but still requires a vatting temperature of 140°F or higher in order to give the correct shade. Absorption spectra of the reduced compound at low temperatures (eg, 40°F) and at high temperatures (eg, 140°F) are markedly different *(1)* (see Fig 1), and this suggests that two distinct forms of the blue leuco compound can

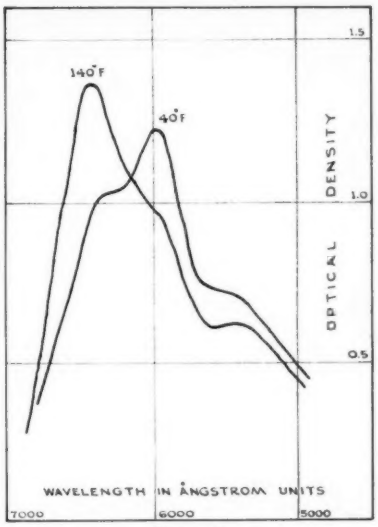
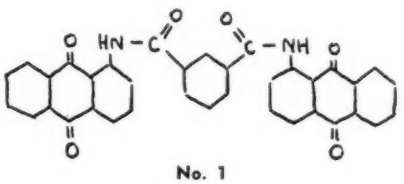
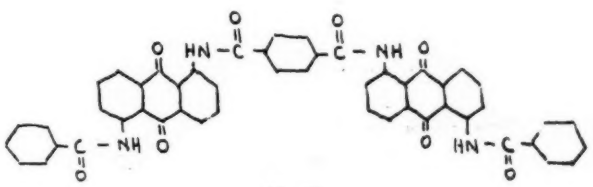
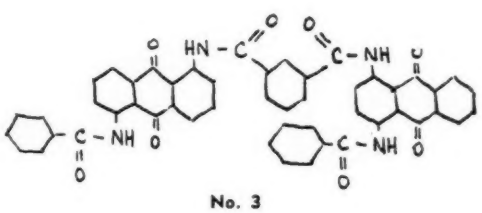


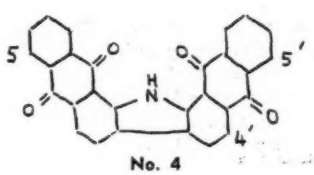
Figure 1
Absorption Spectra of Caledon Yellow GN (leuco forms)

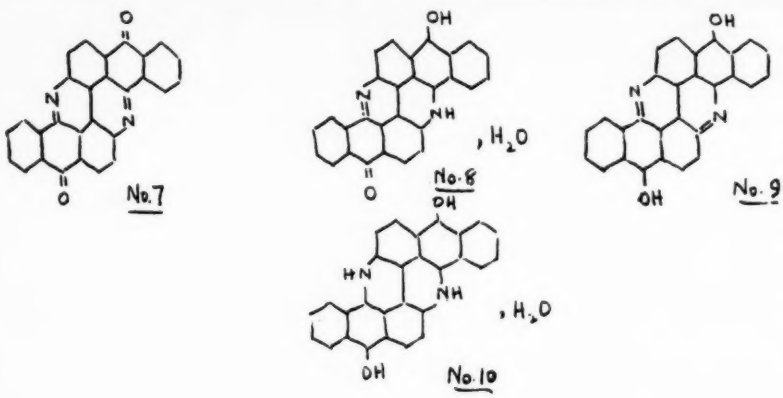
appear in a normal dyebath depending upon temperature. Two possible forms are shown in Nos. 8 and 9 (page P732) and it is believed that the tetrahydro body No. 10 is the brownish vat obtained under certain abnormal vatting conditions. Scholl *(3)* has shown that compounds 8 and 10 are hydrated bodies and suggests that the former is the reduced form of flavanthrone present in the vat at 140°F and higher. Flavanthrone in nylon fades to a green shade on exposure to light for only a few minutes, suggesting that a reduced form of dye is present after exposure. If the flavanthrone is dispersed as a pigment in m-cresol solutions of nylon, light fastness tests show a much higher grading, and it is conjectured that the low light resistance of this dye on nylon is connected with the reducing properties of the fiber and also with its hydrophobic nature and the inability of reduced forms of flavanthrone to become hydrated in this fiber. When flavanthrone is reduced


No. 1


No. 2


No. 3


No. 4



at temperatures around 212-250°F, it gives a stable vat, but in the presence of small amounts of zinc and iron, green and dull shades which do not respond to severe oxidation treatments are obtained. These are associated with the presence of further degradation products, possibly oxanthrone forms of the tetrahydro leuco compound No. 10. Such abnormal shade changes have been traced to the use of galvanized iron buckets for the preparation and storage of print pastes.

OVERREDUCTION ——— Most dyers have experienced overreduction troubles with the indanthrone blues, and some success in the prevention of dye damage has been achieved by the use of sodium nitrite, particularly in continuous dyeing procedures *(4)*. There have been suggestions as to the mode of action of this agent in the dyebath, and these may possibly be true. It is significant, however, that some of the early processes used in the manufacture of indanthrone via the caustic potash fusion of 1-aminoanthraquinone made use of sodium nitrite (and also sodium chlorate) as fusion assistants for the avoidance of dull (overreduced) impurities in the color.

Sodium nitrite can act as a hydrogen acceptor with the ultimate formation of hydroxylamine in the dyebath, thus

$$Na_2S_2O_4 + 2H_2O \rightarrow 2NaHSO_3 + 2H$$

$$NaNO_2 + 2NaHSO_3 \rightarrow HON(SO_3Na)_2 + NaOH$$

sodium oximido-sulfonate

$$HON(SO_3Na)_2 + 2H \rightarrow NH_2OH + SO_3^{--} + Na^{++}$$

hydroxylamine

Thus, sodium nitrite may direct excess hydrogen from attack at the vulnerable —OH groups of leuco indanthrone. The reactions proceeding in the dyebath must be exceedingly complex in the presence of sodium nitrite, and it is a known practical fact that more reducing agents are required to maintain a soluble vat under these conditions. It is doubtful, though just possible, that stable oximes or aldoximes of the indanthrone may be formed, and these could retard the formation of undesirable oxanthrone and anthrone forms of the dye. On the other hand, since there is no experimental proof of such a possibility, the intense side reactions involving the formation of oximidosulfonate and hydroxylamine may be sufficient in themselves to prevent overreduction of the indanthrone blues. Hydroxylamine itself is known to exert a positive protective effect *(4)*.

VAT DYEING IN EUROPE

Turning now from the realms of factual and speculative vat-dyeing chemistry, it may be of interest to some of you to hear a very brief account of the more recent dyeing techniques and practices current in Europe. There is nothing fundamentally new here, but a few points of practical interest are given.

First, it should be stated that the majority of cotton packages, eg, cones, cheese and beams, are still dyed by reduced techniques, and only where very high-twist yarns and dense packages are encountered is the prepigment technique employed; in very pale shades, recourse is made to the use of temperature control and Dispersol VL (ICI). These general remarks are also true of yarn dyeing in hank form.

Large quantities of high-denier continuous-filament viscose rayon yarn is dyed in cake form by the Abbot-Cox technique *(5)*, making use of Dispersol VL (ICI). It is understood that the incidence of cake dyeing is relatively small in the United States, where continuous-filament production is high compared with that of European countries. It is interesting to note, however, that the biggest dyers of rayon cakes in Europe are the rayon manufacturers, and many of these have the opportunity of dyeing rayon which has not been dried after the spinning operation. Several special machines, eg, the Courtaulds cake-dyeing machine and the Enka dyeing machine, have been developed for cake dyeing. By the use of similar prepigmentation techniques for cake dyeing, in the absence of polethanoxy restraining agents, it has been possible to dye viscose cakes at temperatures in the region of 212°F with a saving in time. The Steverlynck and similar static-pressure high-temperature machines are also being worked upon for the dyeing of mercerized-cotton packages with vat dyes at temperatures in the region of 250°F.

The Standfast Molten-Metal continuous dyeing system has met with ready adoption in the vat-dyed piece-goods trade, and the class of goods dyeable by this process has been extended to linens, mercerized poplins, twills and drills, towelling, cotton-pile fabrics of the corduroy and velveteen types, as well as the viscose-rayon/cotton brocade for which the machine was primarily designed. The machine has also been used for continuous warp dyeing on a small scale.

The pad-steam processes of continuous piece-goods dyeing are becoming of increasing importance in Europe and have taken preference over the Williams Unit and the hot-oil process where this type of work is in hand. The pigment-pad → dry → chemical-pad → steam process, as here, has gained preference over both the "wet" process and the acid-leuco process. The latter is now rarely employed, and the shades normally obtainable by this route are being dyed by continuous leuco-ester techniques (eg, with Soledon or Vat-Soluble types of dye). Pigment-padding operations in the presence of caustic soda by the "wet" process of pad-steam dyeing have not proved to be popular, and where the "wet" process has been adopted, this has been done by pigmenting at a low pickup and chemical padding at some 20-30% higher pickup and maintaining a feed of caustic soda and sodium hydrosulfite in solution to offset dilution effects.

REFERENCES

(1) Marshall & Peters, *J Soc Dyers Colourists* 68, 289 (1952).
(2) Fox, *ibid* 65, 508 (1949).
(3) Scholl, *Ber dent Chem Ges* 40, 1694 (1907); 41, 2304, 2316, 2534 (1908).
(4) Zimmerman, Mecco, Fordemwalt & Cooke *Am Dyestuff Reptr* 41, 597 (1952).
(5) Imperial Chemical Industries Ltd, *Technical Circular* (1953), "The Abbot-Cox Process for Vat-Dyeing Goods in Package Form".

• ABSTRACTS •

Pigment Printing and Dyeing

G Bertolina, *Dyer* 111, 991-9, June 25, 1954.

The principle of pigment dyeing and printing is extremely simple, but this very simplicity also gives the explanation of the difficulties that it was necessary to overcome in order to introduce it into industrial practice. Since there is no affinity between pigment and fiber, the fixing of the pigment to the substrate must be achieved by means of suitable binders, that is, resinous materials in which a large number of properties are required that are difficult to find in a single product, but all of which are indispensable. Such properties are principally:

(a) insolubility and non-swelling property in water and in solvents, in order to have high fastness to washing and drycleaning.

(b) ability to form films which adhere to all types of fibers, but which must not be rigid, in order to give high rubbing fastness and at the same time a soft handle.

(c) stability and resistance to heat, light, and chemical agents, so that the binder does not become hard or yellow or lose its adherence to the substrate, owing to chemical transformation or depolymerization.

The products used in pigment dyeing and printing today are subdivided into three large classes: water-in-oil, oil-in-water, and aqueous solution types.

Water-in-oil types. The precondensed resin which acts as binder is dissolved in an organic solvent. The binder (of Aridye 400) is a ureaformaldehyde resin, modified and made oil-soluble, coupled with an alkyd resin; the organic solvent (Varsol 2) is a distillate of petroleum having a high boiling point, with a certain quantity of cyclic hydrocarbons and naphthenes.

The essential novelty of the Aridye process consists of the fact that the thickener is not made up of the normal water-soluble types used in classical printing, but of an emulsion of water in a solution of the binder. From that the name "water-in-oil" comes. A thermal after-treatment is necessary for fixing.

Oil-in-water types. The precondensed resin soluble in organic solvents is emulsified in water beside the dispersion of the pigment. Here we have, besides the aqueous dispersion of the pigment, two or more phases: one continuous in water and one or more discontinuous. For example, in the case of the Orema (Ciba) dyestuffs, besides the aqueous external phase holding the pigment and the catalyst for polymerization, there are two dispersed phases, i e, the emulsion of the binder (thermosetting condensation product derived from urea, thiourea, and formaldehyde, dissolved in cyclohexanol) and the emulsion of the thickener (benzine). For fixing an after-treatment at 120-170°C is necessary.

Aqueous solution types. These are distinguished from the water-in-oil and oil-in-water types by the fact that the substances that act as binders are more or less colloidally soluble in water. Either normal or emulsion thickeners may be used. Polymerization of the resin is obtained by steaming, with the aid of an acid-producing catalyst.

The author gives full details of various printing techniques, including discharge, resist, and flock printing. He also discusses the technique of pigment dyeing, in which, however, it is possible to obtain only light and medium shades, since dark shades give a rather poor handle. He points out that pigment dyeing can be coupled with various finishing processes, e g, crease-resisting, shrinkproofing, and waterproofing.

The paper deserves to be read in its entirety.—WHC

Advanced Features of New Fugitometer

Anon, *Dyer* 112, 61, 63-4, July 9, 1954.

A description, with illustrations, is given of the latest type of the Kelvin Hughes Fugitometer, a British fading lamp similar in principle to the Fade-Ometer.

Samples under test are exposed to radiation from an enclosed flame-type pure carbon arc, from which excess infra-red radiation is filtered by a surrounding water screen.

As the humidity of the atmosphere considerably affects the degree of fading resulting from exposure to light, special provision is made for controlling humidification of the atmosphere surrounding the samples under test, while the ambient temperature of the sample chamber can be adjusted independently.

A newly-designed arc lamp is employed, which can be run continuously for 24 hours without attention. An "hours-run" meter for the lamp is incorporated on the control panel. The frame holding the sample holders is rotated slowly around the lamp.

The humidifier unit operates on a new principle, in which the water is atomized by a high-speed air fan. About 30 gallons of water are said to be evaporated in 24 hours. With ambient conditions of 60°F and 60 percent relative humidity, conditions of temperature and humidity in the sample space can be varied independently from 68°F to 100°F and from 55 percent to 98 percent relative humidity.

The apparatus needs to be connected to a supply of running water and to a water drain. Approximate water consumption is 1 gallon per hour at maximum humidity and temperature.

Several safety features are included, to guard against failure of the arc or the water supply. It is stated that the equipment can be safely left running over night.

The writer points out that fading is a photochemical action and at any given light intensity its rate will increase with temperature. There is therefore an incentive to run at a high temperature, but there are three objections:

(1) There is no guarantee that the fading results at high temperatures will be similar to those at more normal temperatures.

(2) At a high temperature it is almost impossible to maintain a high humidity.

(3) Many materials, especially the rayons and the new synthetic fibers, will themselves discolor if exposed at high temperatures.

It is claimed that in this new Fugitometer samples are kept at very much lower temperatures than in the corresponding American machines.—WHC

Fast Shades on Spun Rayons

P A Holt, *Dyer* 111, 981-9, June 25, 1954.

From the dyer's viewpoint, spun viscose has two outstanding properties: first, its hydrophilic character, i e, its property of water absorption and retention with accompanying swelling; second, a marked tendency to shrink when wet viscose is allowed to dry in the absence of tension.

It was realized at the outset of spun rayon production that good finishes were only obtainable by adopting processes which induced considerable shrinkage in the loomstate cloth, with the result that in dyeing, the beck became the standard machine.

The introduction of continuous methods of vat dyeing in the USA was timely in that it offered the means of satisfactorily dyeing spun viscose to high standards of all-round fastness, with possibilities of the preservation of the shrinkage so necessary in the production of attractive finishes. The size of the domestic market and the vertical integration of the textile industry in USA afforded the long runs per shade that were desirable for continuous dyeing. More recently, experience with the Du Pont pad-steam process and the Standfast machine has demonstrated that short runs per shade can be conveniently dyed.

The author reviews the latest methods of applying vat dyes continuously to

spun rayon. He states that the first essential is that the fabric must be thoroughly desized and scoured at open width.

The Du Pont pad-steam process, the Gen Ray process (General Dyestuff Co) employing the Williams Unit, and the British Standfast Molten Metal process are described in some detail. A more recent continuous system is that employing the Bond dyeing machine (Pittsburgh Coke & Chemical Co). This consists of several pairs of perforated plates between which the cloth passes horizontally, while dyes and chemicals are forced by pressure through the material.

Discussing the application of direct dyes continuously, the author states that the dye is applied by the usual padding technique, and the cloth is then steamed in the wet or dry state for 5-10 minutes. Dyes capable of quick fixation are required. At present the process is limited to pale and medium shades.

After-treatment of direct dyes to increase the fastness is also discussed. The crease-resist finish, which is used on a large scale, frequently reduces the lightfastness of the dyes. However, appropriate after-treating agents (e g, copper complexes or cationic fixing agents) can be added to the resin bath, according to the author, to improve the fastness to light.—WHC

The Problem of Crocking Fastness of Naphthol Dyeings

W Wueterich, *Dyestuffs* 40, 225-37, July, 1954.

The production of a Naphthol dyeing consists of three operations, namely:

1) Impregnation with the Naphthol component.

2) Development with the diazo component.

3) The aftertreatment.

Each of these three operations influences the fastness to crocking of the dyeing.

The author describes all three operations in great detail, and makes the following recommendations.

Precautions to be taken in the Naphthol prepare:

1) Hydroextract the goods as thoroughly as possible after impregnating, to remove all Naphthol not absorbed by the fiber.

2) Use the substantive Naphthols wherever possible, and when the Naphthols of low substantivity have to be used, add salt to exhaust the bath.

3) If possible, dry the goods before coupling with the diazo solution.

Precautions to be taken in the coupling process:

1) Add a suitable binding agent to combine with the alkali carried into the developing bath with the goods.

2) See that the developing bath reacts just slightly acid. All diazo compounds are sensitive to mineral acid; many of them couple completely at pH 3, while with others it is necessary to adjust the pH to the range that is most suitable. Wherever possible acetic acid should be used as the alkali-binding agent when bases are used.

3) Since the Naphthol has a tendency to come off the fiber before it has time to couple with the diazo compound, an addition of salt to the developing bath is recommended.

Precautions to be taken in the after-treatment:

1) Use only soft water for the soaping bath, or if hard water only is available, use a detergent that is unaffected by hardness.

2) Continue soaping for 15 to 20 minutes near the boil until the change of shade that takes place is complete.

3) Remove the after-treating liquor completely from the goods by rinsing at 60-70° C, followed by a second rinse at 40-50° C.—WHC

Nylon Colored with Large Vat Dye Particles

W H Sharkey and F C McGrew, *Textile Research J* 24, 633-6, July, 1954.

Although vat dyes hold a pre-eminent position as colorants for cellulosic fibers because of their excellent washfastness and lightfastness, they have been used very little on nylon. One of the major reasons for this was the generally poor lightfastness of these colors on nylon, a phenomenon that was recognized in the first studies on the dyeing of nylon. More recent studies by Saville demonstrated that only selected blue and green vat dyes are appreciably fast on nylon, and even in these cases undesirable color changes were found to occur in less than 100 hours of Fade-Ometer exposure. These findings were confirmed by Egerton and by Bamford and Dewar in their studies on the tendering of nylon by vat dyes.

It has been reported that the lightfastness of vat dyes on nylon is increased by steaming at pressures above atmospheric pressure. However, this treatment leads to the formation of relatively large vat dye crystals on the fiber surface, due to the migration of color through the softened polymer.

The present study was undertaken to determine the effect on lightfastness of increasing the particle size of vat dyes inside nylon fibers.

Wet-spun yarns were used in this investigation, since large dye particles could easily be incorporated into the fiber by dispersing the color into the polymer solution prior to spinning. In addition, it was found that certain vat dyes, e g, Ponsol Red BN, form large dye particles inside wet-spun nylon fibers by conventional dyeing techniques. Both of these colored wet-spun yarns showed extremely high light-durability, as determined by Fade-Ometer exposures. For example, yarns pigmented with large particles of Ponsol Red BN showed no appreciable change of color with exposures as long as 200 hours; conventionally dyed, wet-spun yarn was stable for 100 hours. On the other hand, melt-spun nylon yarns dyed by conventional methods with the same dye and exposed at the same time faded badly in less than 10 hours.

Three other vat dyes, Ponsol Yellow AR, Ponsol Jade Green, and Ponsol Dark Blue BRA, gave somewhat similar results.

Micrographs (x 500) of the dyed fibers showed that the dye was present as large particles in both the pigmented wet-spun yarn and the wet-spun yarn colored by the usual method, while the dye particles in the dyed melt-spun yarn were too small for detection.

The results of these experiments indicate that a great improvement in resistance to light destruction may be obtained by a 10-fold increase in the average dye particle radii. It is probable that the effect is general for most members of the vat color class, in view of the observations of earlier investigators.

Thirteen references to the literature are cited.—WHC

Variables in Padding Processes

R W Speke, *J Soc Dyers Colourists* 70, 221-6, June, 1954.

Dyeing techniques involving mechanical impregnation of a fabric with a dye suspension or solution, so far as possible at a stage of no affinity, followed by a fixation treatment *in situ*, are becoming increasingly common. This is largely a result of the modern trend towards continuous processes, which are ideally based on the impregnation — fixation sequence. In addition, the enhanced quality of dyeings obtainable by prepadding in conventional dyeing systems, particularly with heavy or tightly woven fabrics, is leading to its adoption in many new fields. Furthermore, the application of a wide range of textile-finishing agents which have little or no affinity for the substrate is most conveniently effected by mechanical impregnation.

The author gives an outline of the salient features governing the design of pad mangles for the application of dyes and finishing agents to woven textiles. He states that the covering of the rollers is one of the most important factors in ensuring efficient operation. Even so, many and varied are the types of coverings encountered — stainless steel, vulcanite, brass, soft rubber, etc — and many combinations of these are found in the individual rollers of mangles. He sum-

marizes the various factors which will influence the choice of covering.

The manipulative variables in the padding of dry fabrics are discussed. These include the preparation of the cloth, the running speed, and the temperature of the pad liquor. Padding assistants, known as "padding oils," should have the following characteristics: high-speed wetting efficiency, freedom from excessive foaming, and no adverse effect on dyes.

The author warns that padding assistants must always be chosen with an eye to the nature of the dyes and the subsequent fixation treatments employed. For example, cationic wetting agents will coprecipitate with anionic dyes, and ethylene oxide condensates will exert a restraining effect during the subsequent fixation of vat dyes.

One of the most common difficulties of this type is encountered with vat dye dispersions in the presence of anionic padding oils. Aqueous suspensions of the highly dispersed forms of pigment vat dyes marketed especially for padding processes may be cracked or flocculated under the combined effects of

1) Presence of padding assistants, the nature and concentration of the assistant being important.
2) Storage, for periods of a few hours only.
3) High temperatures.

The author also discusses the basic principles involved in the repadding of wet fabrics, where it is necessary to avoid an intermediate drying process.—WHC

New Acetate Develops Wool-like Crimp

Anon, *Modern Textiles* 35, 30-1, August, 1954.

Du Pont's new Type C acetate yarn has the property of developing a wool-like crimp when immersed in boiling water. When properly developed, it is stable to laundering and drycleaning. During crimping, some delustering takes place.

This yarn is now being produced commercially in continuous filament form in various deniers. In addition, it can be produced in Du Pont's range of solution-dyed colors.

The ability to crimp has been built into the fibers through special production techniques. As the yarn is shipped, it looks the same as any other continuous filament.

Fabrics are normally crimped in standard dyeing equipment of the enclosed type for 10 to 30 minutes at 205-212°F; the higher temperature greatly increases the crimping action.

The author gives special directions for weaving and knitting these crimped fibers, and lists a number of commercial uses, particularly in pile fabrics.—WHC.

Barotor——

(Concluded from page 711)

The unique advancing action without movement of bars is so simple and yet unexpected that an explanation should be given. Referring to Figure 8 it can be seen that, as each loop of fabric during its rotation comes out of the bath, it picks up a sizable pool of liquor. This weight temporarily pulls downward on the fabric. The loop immediately ahead refuses to give up any slack because it is also weighted down with trapped liquor, but the loop immediately behind is under the surface and is buoyed up by the bath so as to readily yield its slack in response to the forward pull. The result is that each loop, as it approaches the departure from the bath, loses its slack to the loop ahead, and then as it leaves the bath, regains an equal amount of slack from the loop behind, thus bringing about a definite forward shift of all portions of the fabric for each turn of the rotor. The amount of advance can be controlled by varying the amount of slack material loaded on the rotor or by varying the depth of bath so as to change the angle at which the loops emerge. Progressions as great as ten inches per revolution have been obtained, but the usual practice is to limit the advance to one to two inches. Positive shifting of the fabric is absolutely necessary for level dyeing and a one-inch advance per revolution has been found to give a good margin of safety.

ADDED FLEXIBILITY———Conversion from parallel to series loading with new Barotors and those now in use will provide greater flexibility, eliminate the inconvenience of piece length matching, reduce loading time, minimize fabric waste, and permit full capacity batches at all times. The cost of the new auxiliaries is expected to be no greater than the cost of the former loading equipment that is replaced, and the simplified rotor can be employed without change.

This new loading practice has been found to be equally successful with both filament and spun yarn materials. The Barotor from the start has been recognized as being unique in its ability to handle an extremely wide range of fabric constructions, provide open-width relaxed-pressure dyeing, and permit accurate fabric sampling for shade matching without process interruption. Now that there has been added this new feature of random length series loading, the machine is expected to have even greater potential in the dyeing and finishing industry.

REFERENCES

(1) DuPont News Release, August 6, 1952.
(2) DuPont News Release, May 8, 1953.

Bancroft——

(Concluded from page 713)

has resulted in much confusion through the years as many have thought that "quercitron" referred to the bark of the yellow oak rather than to the yellow dyestuff furnished by the black oak. While quercitron was originally used for various yellow shades, its main use was chiefly for the dyeing of brown shades on woolens and mixed fabrics. For over 50 years it was used to impart brown and orange hues to madder and garancin as well as to modify shades produced by cochineal and logwood.

Chevreul was the first chemist to examine the dyestuff obtained from quercitron. He found it to contain, besides a peculiar tannin, a yellow coloring principal to which he gave the name of quercitron. Later investigation showed quercitron to be a glucoside of rhamnose. For many years large quantities of a hydrolysis product of quercitron were also exported from America under the name of flavin. The extensive use of quercitron coloring matter persisted up to World War I and it is used even today in certain parts of the United States for home-prepared colors.

Bancroft's second great contribution was the publication of "Experimental Researches Concerning the Philosophy of Permanent Colours." (See Figure 4). Various practical dyers have felt, however, that it contained too much theory and not enough practical formula. A well-known practical dyer, Edward Parnell, writing in the middle of the last century, had this to say about Bancroft's work:

> "Dr Bancroft's work is little or no use in the dyehouse, being too exclusively theoretical, at the same time that it is unmethodical and full of inexcusable repetitions. It is a complete wilderness of words, often without definite meaning or the least applicability to the subject in hand. It is, in short—as practical men well know—better calculated to mislead than to instruct."

Reading Bancroft's work today, in spite of the fact that many of the dyes mentioned are obsolete, puts the lie to the "practical" Parnell. Bancroft's thoughts and research methods are as reasonable and as practical as the best we use today. As one reads this work it is hard to believe that this honest scientist, writing and experimenting in the field of dyeing in a scientific manner at least 75 years in advance of his times, was the same man who took delight in deceiving and double crossing the country of his birth and the adopted one which had treated him so well.

• Evaluation Program Begins for Cyanoethylated Cotton

In a major step towards materially improving the competitive position of cotton, the first pilot plant for the cyanoethylation of the fiber was officially opened on October 5th at the National Plant of Standard-Coosa-Thatcher Co in Rossville, Ga.

The pilot plant is the result of intensive work by the Institute of Textile Technology and American Cyanamid Co on equipment design and process development for the chemical modification of cotton with acrylonitrile to produce a new textile material. Designed for maximum versatility, this installation permits a complete study of the operation to determine the most economical commercial process.

A minimum of 20,000 pounds of cyanoethylated cotton is scheduled for production at this pilot plant within the next nine months. The new material will be subjected to service tests on a large-scale market evaluation program conducted by the Institute in conjunction with the textile mills constituting its membership.

American Cyanamid Company, a leading producer of acrylonitrile in this country, has supplied and installed the equipment for the pilot plant in facilities made available by Standard-Coosa-Thatcher, a member mill of ITT. The Institute will operate the pilot plant, treating cotton yarns and fiber supplied by its member mills, who will in turn process the cyanoethylated cotton into various end-use products and aid in the market evaluation.

The selection of more than thirty-five different end-use products into which the cyanoethylated cotton will be put is based upon laboratory tests which have shown improved rot resistance, heat resistance, abrasion resistance, chemical resistance, dye receptivity, electrical insulation, as well as yarn and fabric strength.

Further extension of the value of the pilot plant is expected to come from the establishment of the optimum processing conditions for obtaining fibers best suited for specific end-uses at the lowest cost. From the information obtained from the operation of the pilot plant and the market evaluation program, the future of the development can be determined.

The Institute will make the process available to the textile industry in the future through a licensing arrangement based on patent applications by Jack Compton, ITT technical director.

• Celanese Previews Arnel, a New Fiber

A new man-made fiber called Arnel, which is claimed to have a wider potential use than any synthetic yet developed, was previewed at New York's Hotel Pierre earlier this month by Celanese Corporation of American for press representatives.

Developed under the laboratory name of X-100, Arnel was officially introduced as a fiber spun from cellulose triacetate. It is the culmination of a research program covering the past several years directed towards the production of a fiber of balanced performance and price based on the low cost raw material sources of Celanese Corporation. In one volume-priced synthetic it is said to offer, to an unusual degree, the advantages of ease-of-care and wash-and-wear.

Harold Blancke, president of Celanese, stated that commercial production of Arnel has begun. Because the balanced characteristics of the fiber open broad uses in the spun fabric fields, output of staple fiber will be emphasized during the initial period. Plans are to expand production as mill customers develop fabrics which they and Celanese feel will properly perform in the end uses intended.

Of all the fibers yet produced, Mr Blancke said, Arnel achieves the best balance among performance, appearance, versatility, and price. He added that textile mills can now produce fabrics which have the attractive drape and hand of luxury fibers, such as acetate and silk, plus the easy washability, the fast drying, the wrinkle resistance, color fastness, and durable pleatability of the newer synthetics.

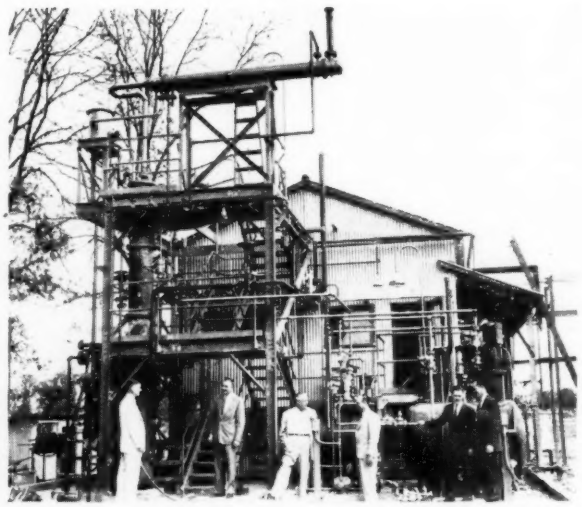
The development and specific properties of the new fiber were discussed and demonstrated by Reiner G Stoll, in charge of applications research and development of the Textile Division.

• New National Aniline Unit on Stream

The maleic-anhydride-fumaric acid unit in the new Moundsville, W Va, plant of National Aniline Division, Allied Chemical & Dye Corporation is now on stream. The new unit is believed to be the world's largest producer of maleic anhydride tablets and fumaric acid crystals. Last February the first unit of this plant was put into operation to produce aniline.

The new Moundsville plant, located on a large tract alongside another Allied Chemical facility, will supplement National's production of dicarboxylic acids and anhydrides at Buffalo.

Right: Exterior view of new cyanoethylation pilot plant at Rossville, Ga.



Below: OFFICIALS PAUSE IN TOUR OF ROSSVILLE PLANT ——(L to r): L A Fluck, manager, Textile Resin Dept, American Cyanamid Co; S H Harris, president, Standard-Coosa-Thatcher Co; L H Hance, president, Institute of Textile Technology; Aubrey A Hobbs, manager, Research Div, Standard-Coosa-Thatcher Co; Jack Compton, ITT technical director; and L C Duncan, manager, Organic Chemicals Div, American Cyanamid Co.

OFFICIALS AT SPECIAL LTI CONVOCATION———A few of the department heads and members of advisory committees of the Lowell Technological Institute are shown with vice president of the United States Richard M Nixon at the recent dedication of Cumnock Hall, new LTI auditorium-administration building.

(L to r): Albert E Chouinard, professor in charge of leather engineering department; E Daniel Lord, J S Barnet and Sons, Inc, leather committee; Dominic Meo, Salem Oil and Grease Co; Francis W White, American Woolen Co, textiles committee; Nathaniel M Mitchell, Barnes Textile Associates, Inc, textiles committee; John Lewis of the paper engineering department; Vice President Nixon; Fritz S Klein, Byron Weston Co, paper committee; LTI president Martin J Lydon; Meith Maeser, United Shoe Machinery Corp, leather committee; Kenneth E Bell, A C Lawrence Leather Co, leather committee; James A Leroy, McLaurin-Jones Co, paper committee; James H Kennedy, chairman of the division of textile manufacturing; William B Wheelwright, paper committee; Gordon Osborne, Warwick Mills, textiles committee.

• LTI Confers Honorary Degree on VP Nixon at Cumnock Hall Dedication

The honorary degree of doctor of science was conferred upon vice president of the United States Richard M Nixon at a special convocation dedicating Cumnock Hall, the new auditorium-administration building, at Lowell Technological Institute, Lowell, Mass on September 28th. About 1000 persons were in attendance to witness the dual ceremonies with Martin J Lydon, president of LTI conferring the degree. Cumnock Hall was named for Alexander Goodlet Cumnock, a founder of the Lowell Textile School and first chairman of its board of trustees.

Greetings were extended to those present by Mr Lydon; Mayor John Janas of Lowell; C Edward Hayes, president of the LTI student council; Prof Lester H Cushing, chairman of the faculty; Kenneth R Fox, former president of LTI and a director of the alumni association; Ralph Lowell, chairman of the LTI board of visitors; and Congresswoman Edith Nourse Rogers. A eulogy on Alexander Goodlet Cumnock was delivered by Senator Leverett Saltonstall. Taking part in the ceremonies of dedication were Mrs Norman E Ditman, daughter of Mr Cumnock; Samuel Pinanski, chairman of the LTI board of trustees; and Governor Christian A Herter.

Marshalls for the processional and recessional were Chapin A Harris for the faculty and Levon M Yacubian for the alumni.

• FMC Opens New York Export Office

Food Machinery and Chemical Corporation has established corporate export offices in New York to represent the company's export lines of industrial and agricultural chemicals and various machinery items.

The new office, at 161 East 42nd Street, combines the export sales group of FMC's chemical divisions under the direction of Robert R Clark, manager of chemical export sales, and a sales engineering staff representing various machinery divisions of the company. At FMC's San Jose Export Division headquarters A W Elwood, coordinator of overseas operations, explained that the new office would provide an eastern contact point where exporters and overseas visitors could secure technical assistance and information on FMC's diversified product lines.

Food Machinery and Chemical Corporation now serves overseas markets with domestically produced goods and products manufactured by subsidiary operations in England, Europe, Australia and South Africa. The company's sales representatives are located in principal cities throughout the world.

• USCS Opens for Chemists, Physicists

The United States Civil Service Commission Eighth Regional Office, Dallas, Texas, has released a special plea for qualified applicants for organic and analytical organic chemist positions involving fundamental and applied research in the utilization of cotton, vegetable oils, fatty acids, terpenes and resin acids, and for physicist positions involving fundamental and applied research in electron optics and the molecular structure and properties of cotton and related textile fibers.

The salary range for these positions is from $3410 to $7040 per annum.

Interested persons may apply at any post office for application forms or obtain them from the Regional director, Eighth U S Civil Service Region, 1114 Commerce Street, Dallas, Texas.

• Monsanto to Build New Plant in New Jersey

Major expansion of facilities for production of phosphate salts and phosphoric acid, including an entirely new Monsanto Chemical Company plant to be located at Kearny, N J, were announced recently by J L Christian, vice president and general manager, Inorganic Chemicals Division.

The Kearny plant will be situated on a deep water channel portion of the Passaic River adjacent to the main line of the Pennsylvania Railroad.

In addition to the Kearny location, the expansion involves added facilities in existing plants at Trenton, Mich, St Louis, Mo, Monsanto, Ill, and Long Beach, Calif. Construction of some of the units already is under way.

Initial facilities at the Kearny site, which are expected to be ready for production by early 1955, will include a unit for the conversion of elemental phosphorus into phosphoric acid, and a plant for the production of sodium tripolyphosphate.

At the Long Beach, Calif, plant, a phosphoric acid unit will be constructed.

Expansion of existing units for production of sodium tripolyphosphate and new facilities to produce tetrasodium pyrophosphate will be undertaken at the Carondelet plant in St Louis. At Trenton, facilities for the production of phosphoric acid will be increased. A new acid burner also will be installed at Monsanto, Ill.

The production of elemental phosphorus at the Soda Springs, Idaho plant will be doubled late this year with the completion of the second electric furnace there. This supply supplements existing production at Soda Springs and at Monsanto, Tenn.

• Hooker's Montague Plant ———The last word in caustic soda and chlorine production

Hooker Electrochemical Company's new $12,000,000 plant at Montague, Michigan, which began operations this past Spring, offers to industry a daily potential source of 153 tons of caustic soda, 140 tons of chlorine, and 1,422,000 cubic feet of hydrogen. Their manufacture is achieved by passing direct current through brine in Hooker Type S-3A electrolytic cells.

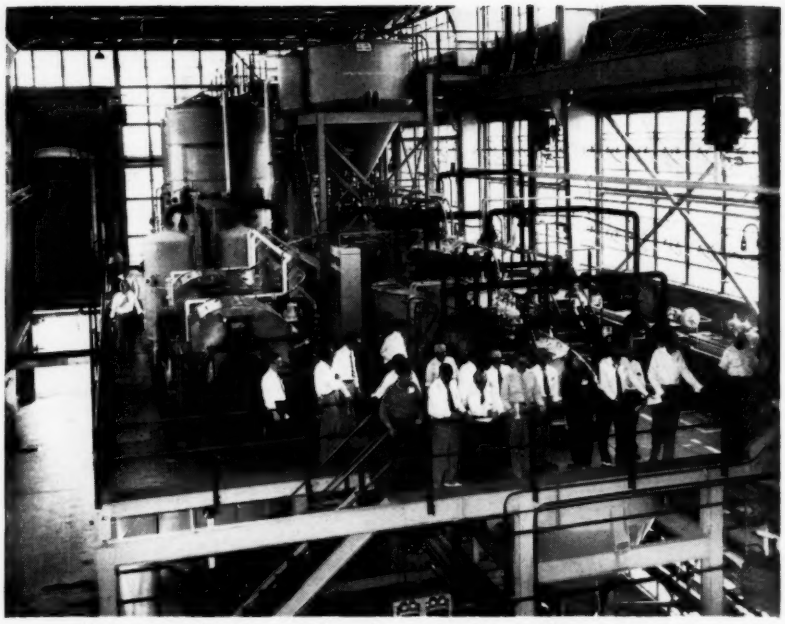
Built by H K Ferguson Co on a 650-acre site in Montague Township, a summer resort area located on White Lake, Michigan, the new installation benefits strategically from the fact that, within a 500-mile radius, 40% of the nation's population and 50% of its industry are concentrated.

Eight major buildings, plus several auxiliary buildings, such as the guest house and recreation hall, occupy 124,000 square feet of floor space. 2,000 tons of structural steel, 300 tons of reinforcing steel, 16,000 cubic yards of concrete, 20 miles of process piping and 8 miles of utility piping were required in their construction.

Other features of the new plant include:

Water Pumping Capacity: Pump house at White Lake contains two pumps — total capacity of 20 million gallons of water per day. In addition, two water wells at brine field and one in the plant area supply substantial amounts of water to make brine and for the potable water supply.

Concrete-lined Equalizing Pond for Effluent Control: Eight million gallons capacity, 300 x 400 feet. The water is 10-12 feet deep. Contains the equivalent of one day's effluent. Water passes over a weir extending the full length of the pond and leaves the pond from two discharge points on the opposite side.



Visitors viewing the caustic soda evaporation building at Montague.

Salt Supply: Located in the Salina salt formation of Western Michigan. 200 acres are set aside as a brine field. The salt is said to be exceptionally pure and is estimated at one million tons to the acre. The salt is in two layers averaging a total of 430 feet in thickness. The three brine wells extend to 3582 feet below the surface of the ground.

Boiler House: Two boilers — combined capacity of 200,000 pounds of steam per hour.

Coal Handling: Capacity 150 tons per hour. Coal can be discharged from cars directly to bunkers in the boiler house or to stock pile.

Smokestack: 225 feet high.

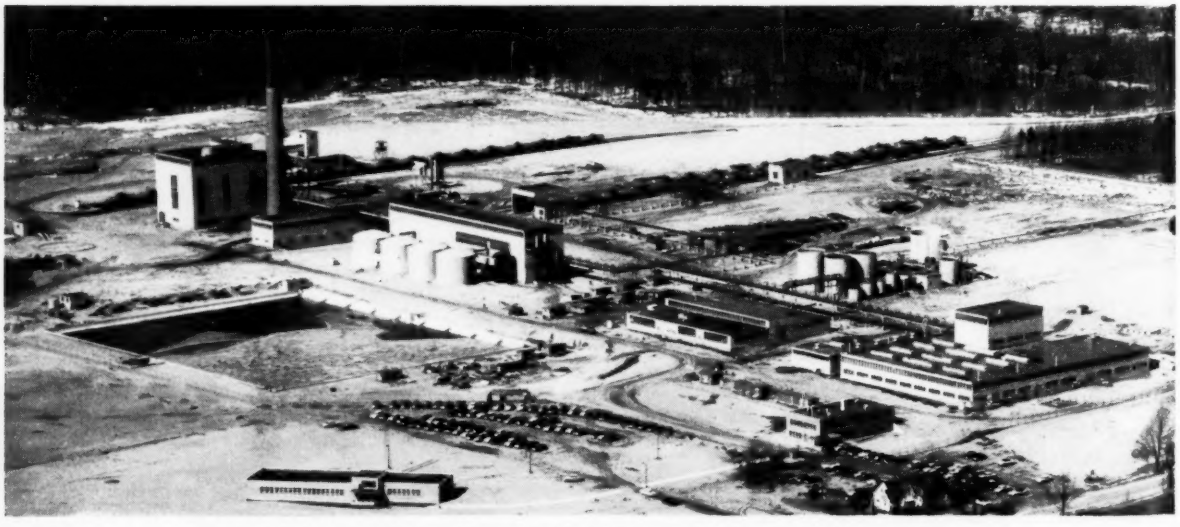
Sewage Disposal Plant: Operate own plant with substantial extra capacity.

Electric Power Input System: Could light a city of 100,000 people.

Automatic Control Devices: Over 300.

Railroad: A two-mile spur was built to the plant by the C & O Railroad.

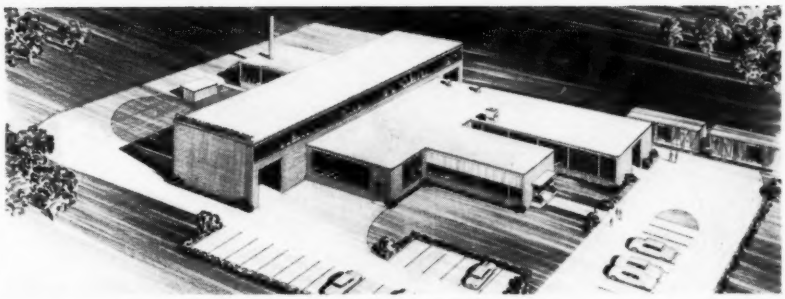
Dockage Facilities: Built to accommodate large barges for caustic soda and for possible incoming raw materials. On June 2, 1954, the first barge shipment of caustic was made from Montague to Hooker's million gallon bulk storage tank at Lake River Terminal, Chicago,



Aerial view of Hooker Electrochemical Company's Montague, Michigan plant.

for distribution from that point. This 400,000-gallon shipment was the first time in 60 years that cargo had been shipped by boat from White Lake — since the lumbering operations of the last century.

Hooker Electrochemical Co, which will celebrate its 50th anniversary next year, now has three plants — the headquarters and main plant at Niagara Falls, N Y, and plants at Tacoma, Wash and Montague. Hooker manufactures about 100 chemicals. Sales are said to currently range over $40,000,000 per year. In addition, Hooker has joint ownership with Detrex Corp in Hooker-Detrex Co, operating plants at Tacoma, Wash and Ashtabula, Ohio, for the manufacture of trichloroethylene.



FUNCTIONAL EFFICIENCY TO MARK STOWE-WOODWARD PLANT IN GRIFFIN, GA———Stowe-Woodward's new plant, when completed about December of this year, will mark a significant advance in rubber-roll production. It is designed solely for the processing of the highly specialized rubber-roll coverings used in textile finishing and papermaking. The new operation will duplicate the facilities of the company's headquarters plant in Newton, Mass, as well as those in the Neenah, Wis, plant, which began operations earlier this year.

NAMES IN THE NEWS

Wicks

Thompson

Gibbons

Medcalf

Aude

Trio

ZENO W WICKS has been appointed general manager of Interchemical Corporation's central research laboratories in New York.

Dr Wicks, who joined Interchemical in 1944, has held a number of posts within the Corporation, at the central research laboratories and several of the divisional laboratories and production units. His most recent position was manager of the commercial research department.

WARREN B THOMPSON, formerly with E I du Pont de Nemours & Co, Inc, Wilmington, Del, has become associated with Rhodia, Inc, New York, as sales representative in the Middle Atlantic states, with headquarters in Philadelphia.

In his new position, Mr Thompson will be responsible for sales of aromatic chemicals and products of Rhodia's Industrial Reodorant ("Alamask") Division, which produces a series of reodorants for industrial odor abatement.

EUGENE F GIBBONS has been added to the sales promotion staff of The Goodyear Tire & Rubber Company's Chemical Division, Akron, Ohio.

For the past two years, Mr Gibbons has been public relations director with Designers for Industry, Inc, independent product development organization of Cleveland, O.

JOHN J RIECK, manager of the Nylon Project of National Aniline Division, Allied Chemical & Dye Corporation, will be manager of the new Chesterfield synthetic fiber plant located in Hopewell and the Bermuda Hundred area of Chesterfield County, Va. A former assistant plant manager at National Aniline's Buffalo, N Y, plant, Mr Rieck has had twenty years' service with the Company.

EUGENE C MEDCALF has been named manager of the newly established Coal Tar Chemicals Section of American Cyanamid Company's General Purchasing Department. The new organization, operating at the Bound Brook, N J, plant, will combine the responsibilities previously assigned to the Coal Tar Chemicals Dept and Company purchasing units. The Section will also be responsible for the disposition of by-products resulting from the processing of crude coal tars and light oils.

Mr Medcalf has been head of the Coal Tars Chemicals Dept since 1952.

* * *

GEORGE K KLAUSNER, formerly with Industrial Rayon Corp, has joined the staff of Cyanamid's Stamford Research Laboratories as a chemical engineer in the Synthetic Fibers Section.

Eastern Color & Chemical Co, Providence, R I, has announced the appointment of GERSON HERMANN as chemist in charge of development and research.

Mr Hermann had previously been employed by the Alrose Chemical Co, Division of the Geigy Chemical Corp, in various capacities as textile research chemist, chief control chemist and plant chemist for a period of 9 years.

ROBERT M AUDE has been appointed plant manager of Heyden Chemical Corporation's Garfield, N J, plant, and EDWARD TRIO, manager of Heyden's Fords, N J, plant, effective October 1st.

Mr Aude has been manager of the Fords plant since 1953, while Mr Trio has been operating superintendent at Fords since 1952.

ERNEST H VOLWILER, president of Abbott Laboratories, North Chicago, Ill, and chairman of the board of directors of the American Chemical Society, was awarded the Chemical Industry Medal of the American Section of the Society of Chemical Industry at a meeting of the section at the Palmer House, Chicago, on Tuesday evening, October 12.

WILLIAM F GEORGE, New York district sales manager for Hooker Electrochemical Co, retired at the end of September in accordance with the company's retirement policy. He is being replaced by HARRIS C MILLER, who has been assistant New York district sales manager.

A dinner on September 17 at the Niagara Falls Country Club at the end of the annual three-day Hooker sales conference was highlighted by a testimonial to Mr George. His colleagues presented him with two large silver candelabra and a silver fruit bowl of matching design as a token of their affection.

Mr George was first employed briefly by Hooker in 1912 purchasing construction materials for a plant expansion. He returned in 1915 in a similar position for another expansion phase. When this was completed he was transferred to the sales department in New York City. He left the company in 1916 to form the W F George Chemical Co. He was later co-publisher of Chemical Industries, *the forerunner of the present* Chemical Week *magazine.*

He returned to Hooker in 1942 when he was put in charge of the New York sales office. He recently completed two years in office as president of the Chemists Club of New York and is a charter life member of the Salesmen's Association of the Chemical Industry.

The Allied Chemical & Dye Corporation Fellowship in Chemistry at the University of Michigan for the year 1954-55 has been given to ARNOLD C SCHOENTHALER, majoring in organic chemistry.

CLAUDE A HANFORD, Pharmaco, Inc, was elected the 58th Chairman of the Drug, Chemical and Allied Trades Section of the New York Board of Trade (DCAT) at its 64th Annual Convention held at Pocono Manor, Pennsylvania, September 23-25. Other officers elected were vice chairman —— *SYDNEY N STOKES, Northeastern Division, Merck & Company, Inc;* treasurer —— *DAVID HAYDEN, R P Scherer Corporation;* counsel —— *JAMES G FLANAGAN, S B Penick & Company, and* secretary —— *HELEN L BOOTH.*

The following alignment of personnel has been announced by SAMUEL S HARKAVY, director, Paper Dept, Geigy Dyestuffs, Division of Geigy Chemical Corp:

JOHN J deJONGH will continue to call on the New England accounts and will also cover parts of New York state. In addition he will act as technical aid and sales consultant to G A WILKIE and R W REDSTON in the Quebec area of Canada.

F G THWAITES will continue to service the mills in the Ontario and Western areas.

DUDLEY J NIXON will cover the paper mills in the Philadelphia area, and will also continue to cover accounts in the metropolitan New York area. In addition he will act as technical aid and sales consultant to L C VOSS in the Ohio area.

HERMAN H GOTTSLEBEN will continue to call on accounts in Wisconsin, Minnesota and Michigan. He will be assisted by WILLIAM A LUDKE, who will look after details and shipping at the Appleton, Wis warehouse.

DANIEL P FESSIA, who will operate out of New York, will act as technical aid and sales consultant for the Charlotte, N C, and Chicago, Ill branches.

WALTER KENOWSKI heads laboratory service in New York.

Three new members were elected to the board of directors of Hercules Powder Co at a regular meeting of the board last month in Wilmington, Del. They are: L W BABCOCK, director of personnel for the company since 1945; JOHN E GOODMAN, treasurer; and ERNEST S WILSON, director of engineering since 1947.

HERBERT H HATCH, president of Hatch Textile Research, Inc, New York, marked his 46th anniversary in the textile business last month.

The following are now representing Haas Miller Corp, Philadelphia, in the South: ARNOLD ISAACS, JOHN LEE, NORMAN McCALL, and JAMES FORTUNE.

Mr Isaacs will represent the firm in a technical capacity in North Carolina, while Messrs Lee, McCall and Fortune will handle sales in North Carolina, South Carolina, and Georgia and Alabama, respectively.

New appointments to the faculty of the Philadelphia Textile Institute include WILLIAM H HENRY, JR, instructor in English; RAYMOND J LODISE, instructor in chemistry; DONALD J MARVA, instructor in physics; and HORACE NEILD, instructor in fabric design and structure.

GENERAL CALENDAR

AMERICAN INSTITUTE OF ELECTRICAL ENGINEERS
Southern Textile Conference—Nov 4-5, North Carolina State College, Raleigh, N C.

AMERICAN SOCIETY FOR QUALITY CONTROL
Annual meeting, Textile Div—Jan 27-29, Clemson College School of Textiles.

DELTA KAPPA PHI FRATERNITY
1955 Annual Convention—April 29-30, Lowell, Mass.

DEUTSCHE GESELLSCHAFT FÜR CHEMISCHES APPARATEWESEN
Achema XI—May 14-22, Frankfurt am Main, Germany.

DRYSALTERS CLUB OF NEW ENGLAND
Oct 29, Jan 24, April 29 (Hotel Vendome, Boston, Mass); June 24 (Outing—Wachusett Country Club, W Boylston, Mass.)

THE FIBER SOCIETY
Spring Meeting—May 4-5, 1955, Alabama Polytechnic Institute, Auburn, Alabama.

Fall Meeting—Sept 8-9, 1955, Massachusetts Institute of Technology, Cambridge, Mass.

INTERNATIONAL TEXTILE EXHIBITION
2nd Exhibition—June 25-July 10, 1955, Brussels, Belgium.

INTERNATIONAL WOOL TEXTILE RESEARCH CONFERENCE
September, 1955—Sydney, Melbourne, and Geelong, Australia.

MANUFACTURING CHEMISTS' ASSOCIATION
4th Semi-annual meeting and Winter Conference—Nov 23, Hotel Statler, New York, N Y.

NATIONAL ASSOCIATION OF HOSIERY MANUFACTURERS, KNITTED OUTERWEAR ASSOCIATION, THE UNDERWEAR INSTITUTE
42nd Knitting Arts Exhibition—April 25-29, Convention Hall, Atlantic City, N J.

NATIONAL COTTON COUNCIL OF AMERICA
Annual Meeting—Jan 31-Feb 1, Hotel Shamrock, Houston, Tex.

Cotton Research Clinic—Feb 16-18, Pinehurst, N C.

NATIONAL INSTITUTE OF DRYCLEANING
4th Educational Conference—Dec 5-10, Silver Spring, Md.

NEW YORK PIGMENT CLUB
Nov 10, Dec 9, Jan 15.

TEXTILE RESEARCH INSTITUTE
25th Annual Meeting—March 10-11, Hotel Commodore, New York, N Y.

nnounced
eigy Dye-

New Eng-
ork state.
sultant to
c area of

lls in the

s in the
counts in
ill act as
the Ohio

ll on ac-
e will be
er details

ew York,
Charlotte.

in New

directors
oard last
ABCOCK,
JOHN E
irector of

Research,
he textile

ler Corp,
HN LEE,

capacity
rtune will
Georgia

phia Tex-
instructor
chemistry;
HORACE

HOSIERY
D OUTER-
UNDER-
April 25-29,
CIL OF
Hotel Sham-
Pinehurst,
YCLEAN-
5-10, Silver
TE
9-11, Hotel